SAMPLING AND ANALYSIS PLAN--QUALITY ASSURANCE PROJECT PLAN FOR SITE INVESTIGATIONS AND RCRA FACILITY INVESTIGATIONS AT THE FORT CHAFFEE FACILITY, FORT CHAFFEE, ARKANSAS

by Phillip D. Hays and Gregory P. Stanton

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ABSTRACT

The quality assurance project plan (QUAPP) component of the Fort Chaffee Sampling and Analysis Plan is designed to document the quality assurance and quality control procedures that will be used during data collection and data analysis for environmental studies to be undertaken by the U.S. Geological Survey at the Fort Chaffee facility. The QUAPP will ensure that the data generated as a result of the Resource Conservation and Recovery Act facility investigations and site investigations are of sufficient quality and quantity to support investigation objectives. This QUAPP address the major elements outlined in the U.S. Environmental Protection Agency Consent Order for the Fort Chaffee facility.

Aspects of quality assurance covered by the QUAPP include: (1) definition of quality assurance oversight and responsibilities for the U.S. Geological Survey, the U.S. Army, and for regulators, as well as defining the responsibilities of the contract laboratory; (2) definition of quality assurance objectives and characteristics of data quality; (3) description of the sampling and analysis quality control procedures to be used for the Fort Chaffee project and delineation of the quality control sample regiment to be applied; (4) description of data reduction, validation, and reporting procedures for the field and laboratory settings; and (5) procedures for assessing data and implementing corrective actions. The QUAPP must meet the challenge of changing regulatory requirements, technical improvements, and unforeseen circumstances to be encountered at the varied sites that must be addressed. Therefore, improvements must be expected to be incorporated over time to meet changing requirements and take advantage of advancing technologies and approaches.

1.0 PROJECT DESCRIPTION

1.1 Objectives of Quality Assurance Project Plan

The objective of the quality assurance project plan (QUAPP) is to document the quality assurance and quality control (QA/QC) procedures that will be used during both data collection and data analysis. The QUAPP will ensure that the data generated as a result of the Resource Conservation and Recovery Act (RCRA) facility investigation (RFI) are of sufficient quality and quantity to support the site investigation and RFI study objectives. The terms quality assurance (QA) and quality control (QC) are often used interchangeably. For this project, QA refers to an integrated and overall program of controls developed to address, document, and certify the quality of data. QC refers to specific steps and controls to monitor the measurement process. This QUAPP addresses the major elements outlined in the U.S. Environmental Protection Agency (EPA) Consent Order for the Fort Chaffee facility.

The QUAPP QC limits may be updated as the project evolves and as relevant information becomes available; in addition, the QUAPP may be reviewed and out-moded elements may be altered and updated on an annual basis.

The overall objectives of site investigations and RFI studies to be conducted at Fort Chaffee, Arkansas are: (1) determine or confirm if a release of hazardous materials has occurred at facility Solid Waste Management Units (SWMU's), (2) determine the nature and extent of any potential threats posed where the release of hazardous substances has occurred, and (3) evaluate proposed remedies. After following the RFI process, a cost-effective remedy, which mitigates threats and provides protection for public health and the environment, will be selected for implementation at each of the SWMU's where a public health or environmental risk exists during the course of subsequent Corrective Measures Study and Corrective Measures Implementation actions that follow the RFI.

1.2 Project Background

The U.S. Army at Fort Chaffee, represented by the Fort Chaffee Environmental Branch, has received a draft RCRA Administrative Order of Consent 3008(h) from the U.S. Environmental Protection Agency, Region VI, concerning investigative and corrective action at Solid Waste Management Units (SWMU's) located on the Fort Chaffee facility. The Order of Consent exercises EPA regulatory authority with the purpose of ensuring that investigative and corrective activities are designed and implemented in order to protect human health and the environment. The U.S. Geological Survey (USGS) will provide technical assistance to the Fort Chaffee Environmental Branch for support of the U.S. Army Environmental Program at the Fort Chaffee facility. Support will be oriented toward such program and functional areas as Environmental

Protection Agency Administrative Order of Consent and Corrective Action Plan compliance, Installation Restoration Program activity, data management, water quality and site hydrogeology characterization, and other Fort Chaffee environmental concerns. This assistance will consist of environmental program design and management, environmental analysis, site characterization, hydrogeologic and hydrologic studies, chemical characterization, quality assurance and quality control, research and development, instruction, training, and other technical activities relevant to environmental support and falling within the scope of USGS expertise in water and environmental science.

A summary description of Fort Chaffee, local hydrogeology, and previous studies are given in the current conditions report, work plan, and field sampling plan (FSP).

1.3 Data Collection Activities

To characterize the nature and extent of hazardous substances on Fort Chaffee, the following field activities will be performed as needed at individual SWMU's in accordance with the FSP.

- Drilling and installing monitor wells.
- Drilling test holes and soil borings.
- Sampling and analyzing ground water from existing and new wells.
- Performing in-situ aquifer test at selected sites.
- Performing geotechnical and chemical analysis on the soil samples collected during the drilling program.
- Collecting and interpreting borehole geophysical logs.
- Performing surface geophysical surveys.
- Sampling and analyzing surface-water samples from selected sites.
- Collecting and analyzing biological samples.
- Collecting and analyzing appropriate QA/QC samples.
- Collecting samples during ongoing treatment as necessary.
- Collecting any treatment-specific data required.

2.0 QUALITY ASSURANCE OVERSITE

2.1 Quality Assurance Responsibilities

2.1.1 U.S. Army

The U.S. Army will monitor progress of the investigation through any field and laboratory audits they wish to conduct, reports of corrective actions, if any, and progress reports submitted by the USGS. The United States Army will respond to comments from the regulators (section 2.5.2) and provide oversight to the USGS.

2.1.2 Regulators

The EPA and Arkansas Department of Pollution Control and Ecology (ADPCE) are the regulators. The EPA has instigated the consent order for the Fort Chaffee facility requiring investigation of facility SWMU's.

2.1.3 U.S. Geological Survey

The USGS is the lead contractor; the USGS provides scientific and technical support to the U.S. Army Fort Chaffee Environmental Branch and is responsible for providing all project work plans and ensuring that the investigation is carried out in accordance with applicable plans. The primary responsibility for project quality rests with the USGS. The Project Chief and Quality Assurance Manager will have direct responsibility for field QC. The field leader will report to the Project Chief manager. Corrective actions may be started onsite by field personnel. These actions will be documented. The project QA/QC officer may also initiate corrective action and must carefully document the rationale and the action. The project QA/QC officer will inform the field leader of any required corrective action.

2.2 Responsibilities and Authorities of Contract Laboratory Personnel

2.2.1 Contract Laboratory Quality Assurance Director

The QA effort within the laboratory will be directed by the QA director whose responsibilities include:

- Developing and implementing a QA program that ensures all data generated in the laboratory are scientifically sound, legally defensible, and of known precision and accuracy.
- Developing and implementing new QA procedures within the laboratory to improve data quality.

- Conducting audits and inspections on a regular basis, reporting the results of those audits, and applying corrective actions as needed to ensure compliance with the QUAPP.
- Coordinating the distribution of performance evaluation samples on a routine basis, evaluating the results of those samples, and applying corrective actions as needed to ensure that the laboratory is able to generate data that meet or exceed the data quality objectives defined in the QUAPP.
- Establishing a database that accurately reflects laboratory performance.
- Coordinating certification programs within the laboratory.

Each divisional QA department in the laboratory will be managed by a divisional QA manager whose responsibilities include:

- Implementing laboratory QA policies.
- Monitoring the implementation of the QUAPP within the laboratory to ensure complete compliance with QA objectives.
- Conducting internal audits to identify potential problems and ensure compliance and written procedures.
- Performing statistical analyses of QC data and establishing databases that accurately reflect laboratory performance.
- Prescribing and monitoring corrective actions.
- Monitoring the preparation and verification of analytical standards.
- Assisting chemists in the writing of operating procedures.
- Reporting the status of the laboratory QA program to the management and the Project Chief with formal and informal communications.
- Maintaining records and archives of all QC data, performance evaluation sample results, audit comments, and inquiries concerning data quality.
- Assuring that the laboratory staff has access to current operating procedures.

•Informing the Project Chief of improvements in laboratory capabilities, the contract laboratory standard operating procedures or Quality Assurance Program Plan for Environmental Analysis documents so that any improvements may be reflected in the Project QUAPP and sampling and analysis plan.

•Monitoring laboratory performance in the areas of holding times, turn-around times, and meeting contractual obligations.

•Assisting in the preparation of the QUAPP.

•Serving as a member of the QA committee.

•Auditing subcontractors.

2.2.2 Laboratory Divisional Manager.

The laboratory divisional manager is directly responsible for ensuring that all employees in that division are complying with the QUAPP. This includes:

•Actively supporting the implementation of the QUAPP within the laboratory.

•Maintaining accurate operating procedures and enforcing their use in the laboratory.

•Maintaining a work environment that emphasizes the importance of data quality.

•Ensuring all resources of the laboratory are available on an as-needed basis.

•Performing overview and approval of final analytical reports.

2.2.3 Laboratory Sample Control Group Director.

The laboratory sample control group director is responsible for:

•Receiving and inspecting the incoming sample containers (section 5.2).

•Verifying and maintaining chain of custody and correct procedures.

•Advising the divisional manager of sample receipt and inspection.

•Controlling and monitoring access/storage of the samples, standards, and extracts.

2.2.4 Responsibilities of Program Manager

•Assist in setting new monitoring programs, including defining the analytical scope of work in terms of the applicable regulatory requirements, required tests, matrices, analyte lists, etc.

•Assist in the preparation of the QUAPP.

•Communicate project requirements to project administrator and other laboratory staff.

•Provide final review of laboratory reports.

•Respond to technical inquiries from the Project Chief.

•Report the status of the laboratory QA program to the Project Chief.

•Inform the Project Chief of improvements in laboratory capabilities, standard operating procedures, or Quality Assurance Program Plan so that any improvements may be reflected in the QUAPP and sampling and analysis plan.

2.2.5 Responsibilities of Program Administrator:

•Receive bottle orders from USGS field personnel and ensure that a sufficient number of the correct sample containers are available to field personnel when needed.

•Oversee the sample log-in process, resolve any discrepancies with sample shipments, and release samples for analysis after verifying that the proper analytical tests have been assigned.

•Monitor the status of active projects to ensure that due dates are met.

•Notify USGS of problems affecting data quality so that a prompt resolution can be reached.

•Prepare final analytical reports in accordance with project requirements.

2.2.6 Access Control

The laboratory will be a secure, controlled access facility. All visitors are required to sign in at the front desk. These visitors will be accompanied by a laboratory employee at all times. During non-business hours, security will be provided by a 24-hour alarm system.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT

3.1 Quality Assurance Objectives

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide results, which are technically and legally defensible, and ensure that data generated are suitable and valid to meet the objectives of the site investigations and RFI's for Fort Chaffee. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of equipment, and corrective action are described in other sections of the QUAPP and the FSP. This section addresses the analytical levels for the ultimate use of the data and the general characteristics of data quality as related to the parameters of precision, accuracy, representativeness, comparability, and completeness.

The Data-Quality Objectives (DQO's) developed for this project are designed to address the different uses of the RFI data to be collected. Data quality is the degree of certainty inherent in a database with respect to precision, accuracy, reproducibility, completeness, and comparability. The DQO's are both qualitative and quantitative specifications for the quality of data required to support RCRA corrective action activities at Fort Chaffee. These activities include the field screening, interim measures, site characterization, and health and environmental assessment phases of the RFI. The DQO's also support the evaluation and selection of corrective measures during any corrective-measure studies needed. QC procedures are outlined in Appendix F.

3.1.1 Analytical Levels and Data Uses

The five analytical levels that address data uses and the QA/QC effort are:

I--Field screening or analysis using portable instruments (largely qualitative).

II--Field analyses using more sophisticated portable analytical instruments (more quantitative).

III--Analyses performed in an offsite analytical laboratory using standard, documented procedures.

IV--Contract laboratory Appendix IX analytical services or laboratory procedures with equivalent QA/QC procedures.

V--Nonstandard analyses and procedures.

The data collection effort will be designed to support the ultimate uses of the data. Data uses and the appropriate analytical levels are listed in the table below.

Data use	Appropriate analytical level	Matrix
Site characterization	I, II, III	Soil, ground water, ecology
Worker health and safety	I, II	Soil ground water, air
Risk assessment	III, IV	Soil, ground water, ecology
Evaluation of remedial alternatives	II, III, IV	Soil, ground water, ecology
Engineering design of remedial alternatives	II, III, IV	Soil, ground water, ecology
Baseline data	I, II, III, IV	Soil, ground water, air, ecology
Disposal of wastes	II, III, IV	Soil ground water

3.2 Characteristics of Data Quality

Data "quality" may be most definitively assessed through evaluation of the precision, accuracy, representativeness, comparability, and completeness of the data. A short description of precision, accuracy, representativeness, comparability, and completeness parameters is provided in the following sections. The criteria for evaluating precision, accuracy, and completeness are outlined in section 12 of this document. A short description of each parameter follows along with the field and laboratory general procedures.

3.2.1 Precision

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Precision of field sampling methods will be evaluated by:

- Laboratory analyses of samples collected in duplicate.
- Duplicate measurement of hydrologic properties.

Precision of laboratory analytical data will be evaluated by:

- Laboratory control samples (LCSs)--replicated analyses of analytes shall be within laboratory-established control limits. An LCS pair also is referred to as Duplicate Control Sample (DCS).
- Matrix spike duplicates--agreement between duplicate analyses of organic spiked analytes shall be within the relative percent difference (RPD) limits specified in SW 846 (U.S. Environmental Protection Agency, 1986b) or laboratory-established limits, whichever is most conservative.

•Matrix duplicates--agreement between duplicate analyses of environmental samples shall be within the RPD limits listed in Appendix B.

3.2.2 Accuracy

Accuracy is the degree of agreement of a measurement with an accepted reference or true value, usually expressed as the difference between the two values, or the difference as a percentage of the reference or true value. Accuracy is a measure of the bias in a system. Accuracy of field measurements will be evaluated by:

•Conducting field measurements of blind samples.

•Proper calibration of equipment as described in section 6.1 of this document, along with periodic calibration checks.

Accuracy of laboratory analytical data will be evaluated by:

•Using primary calibration standards obtained from the National Institute of Standards and Technology, EPA repository, or reliable commercial sources.

•The use of audit samples--laboratory performance on EPA water supply and water pollution audit samples must be such that certification is maintained.

•The use of surrogate spikes--recovery of organic surrogate analytes shall be within the laboratory-established average recovery of the surrogate analyte as specified in Appendix B.

•DCS--recovery of analytes shall be within the limits specified in Appendix B of the laboratory-established average recovery of the analyte. For multianalyte samples, 80 percent of the analytes must be within control limits. Control limits are established by the laboratory based on historical method performance (Appendix B).

•Matrix spikes--recovery of spiked organic analytes shall be within QC recovery limits specified in SW 846 (U.S. Environmental Protection Agency, 1986b) and as listed in Appendix B. Laboratory-established limits may be used if more conservative than the method-specified limits.

3.2.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent population characteristics, parameter variation at a sampling point, a process condition, or an environmental condition. Representativeness of field data will be evaluated by:

- Use of standard scientific methods of measurement and sample collection as specified in the FSP.
- Documentation of reasons for use of nonstandard techniques.
- Adherence to chain-of-custody procedures.

Representatives of laboratory analytical data will be evaluated by:

- Use of preservation techniques (including chilling during shipment).
- Holding times prescribed in SW-846 or 40 CFR 136 (U.S. Environmental Protection Agency, 1986a), as applicable, shall be adhered to (refer to Appendix D).
- Field and laboratory blank analyses.
- Matrix spikes (determine the presence of matrix effects).

3.2.4 Completeness

Completeness is a measure of the amount of data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Completeness of field data will be ensured by:

- Recording all measurements and observations in a notebook.
- Recording and documenting all deviations from standard operating procedures (SOPs).

Completeness of laboratory analytical data will be evaluated by:

- Checking each data set (batch) to ensure they contain all QC check analyses and verifying precision and accuracy for the analytical protocol.
- Checking all batches to ensure they contain all field and trip blank analyses.
- Recording all pertinent dates (dates received, extracted, and analyzed).
- Performing and documenting all requested analyses or providing the reason for nonperformance.

3.2.5 Comparability

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability of field measurements will be evaluated by:

- Use of standard, documented methods, recognized by the scientific community.
- Use of consistent reporting units.

Comparability of laboratory analytical data will be evaluated by:

- Use of standard methods, generally EPA methods; initial project detection limits are listed in Appendix C.
- Use of consistent reporting units.

4.0 SAMPLING PROCEDURES

4.1 Sample Locations

Sample locations will be detailed in the individual SWMU discussions in the work plan. In general, soil samples will be located in a grid to encompass the suspected area of contamination. If samples from the perimeter of the grid are found to be contaminated, the sampling grid will be expanded. This procedure will be used until the area of contamination has been delineated. Soil samples generally will be collected, at intervals, from the surface to the water table. Intervals of soil samples suspected to be contaminated because of color, odor, or field instrument reading, will be sampled even if that interval was not included in the work plan. Background samples will be collected outside the zone of contamination.

Ground-water monitoring wells generally will be located at the site and downgradient of the site. Before monitor wells are drilled, one or more test holes will be drilled to obtain information on the flow directions, lithology, and physical properties of the aquifer material. The test holes may be completed so that they can be used as monitoring wells at a later date. An upgradient well will be used for background information. Additional wells will be drilled, if required, to determine both areal extent and depth of contamination.

New locations for soil borings, wells, and test holes will take into consideration data from previous investigations and conceptual models. Additional information from aerial photographs, soil-gas surveys, and geophysical surveys will be used when applicable. The work plans for each SWMU will detail any additional procedure.

Surface-water sampling sites are chosen on the basis of the location of potential sources of contamination. Generally, a site near the contamination source will be chosen to evaluate the potential of the surface water being contaminated. Sampling sites upstream will be selected for background analysis. Downstream sites will be selected to evaluate the fate and transport of contaminants.

4.2 Sample Collection

A detailed description of sampling activities is provided in the FSP. The FSP presents the sampling methodologies that will be used during each activity to ensure that data are representative of the environmental conditions. Site-specific details will be provided in the work plans for each SWMU. Care will be taken to ensure that the preservatives (section 4.3) contained in the sample containers are not washed out during sampling.

4.3 Sample Preservation and Storage

Water samples will be preserved in the field. The required preservation reagents (Appendix D) may be added to the sample containers by the laboratory before the containers are shipped to the project site, or they may be added in the field. Soil samples will be collected in containers provided by the contract laboratory. Activated carbon and particulate filters from any air sampling will be placed in containers provided by the laboratory. Sediment and sludge samples will be stored in containers specified by the type of analysis (Appendix D). In general, all required samples will be collected, placed in a transport cooler to be chilled to 4 °C, then transported to an overnight shipping collection point for shipment. Upon receiving the transport cooler, the laboratory will record the temperature on the chain-of-custody sheet.

4.4 Sample Transport

To ensure that holding times are met, all samples will be shipped via overnight express to the laboratory for analysis. Holding times, sample containers, minimum volumes, and preservatives are listed in Appendix D. Saturday or holiday arrival of samples will be arranged with the shipping agency and the receiving laboratory.

4.5 Sample Records

Records of sampling events will document the location and time of sample collection, calibration information, and any QA/QC information. These records will be kept in bound field notebooks with nonrefillable pages. Chain-of-custody procedures and methods are discussed in detail in section 5.

5.0 SAMPLE CUSTODY

The purpose of sample custody is to document and maintain the integrity of all samples during collection, transportation, and laboratory operations. A record will be maintained that includes all transactions concerning each sample.

5.1 Field Sampling Operations

An adequate number of samples will be collected to characterize site conditions and to perform the laboratory analyses. The field work team leader personally is responsible for the care and custody of the samples until they are properly transferred or dispatched to the laboratory.

The analytical services request form (ASR) will include a chain-of-custody record. The form will be completed for each sample set at the time of sampling. The form is a self-carbon type containing two sheets. One copy will be kept by the field team leader and the other copy will accompany the samples shipped to the laboratory.

Each sample container will be clearly labeled at the time of collection using adhesive-backed labels and water-proof ink. As a minimum, the information on the labels will include:

- a unique sample ID number or name,
- date of sample collection,
- time of sample collection,
- signature of the sample collector, and
- preservative used.

The sample containers will be sealed in plastic bags to protect the labels from water damage and to ensure that the containers are not in direct contact with the chilling medium. The bagged samples will be placed in an ice chest and chilled to 4 °C per section 4.3. The associated ASR will be sealed in a plastic bag and accompany the samples in the ice chest.

5.2 Laboratory Operations

Quantera Environmental Services laboratories will act as the contract laboratory and conduct analyses of samples requiring an analytical level specifying a contract laboratory. In obeisance with the EPA Draft Consent Order, analyses will focus primarily on 40 CFR Part 264 Appendix IX constituents (other parameters will be analyzed on a site specific basis, as determined by the USGS project chief). Dioxins and furans will be analyzed by Quantera, Sacramento. All other analyses will be performed by Quantera, Denver. The contract laboratory will follow the most recent internal Quality Assurance Management Plan. Samples received by the laboratory's

sample control group (SCG) will be checked carefully for label identification, chain-of-custody, and any discrepancies. Photographs will be taken to document discrepant conditions, wher physical damage or tampering is apparent. Other items of interest include: air bubble in samples of volatile organic compounds, incomplete sample labels, incomplete paperwork, discrepancies between sample labels and paperwork, broken or leaking containers, and inappropriate caps or bottles. Any discrepancies will be brought to the attention of the project chief and resolved prior to starting analyses.

Each sample is assigned a unique laboratory identification number through a laboratory information management system (LIMS), which stores all identifications and essential information. The LIMS system tracks the sample from storage through the laboratory system until the analytical process is complete and the sample is back in the custody of the SCG for disposal or return to the client. Access to the laboratory is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

5.3 Final Evidence Files

A file will be created for the Fort Chaffee project at the laboratory. It will contain all documents associated with the project. This will include correspondence, chain-of custody records, raw data, copies of laboratory notebook entries, and a copy of the final laboratory report. During any month in which there is laboratory or data generating activity relating to the Fort Chaffee project, an update of the file will be delivered to the USGS Project Chief. After completion, all the records are given to the document custodian who inventories the file, checks for completeness, and places the file into the document archive at the laboratory.

6.0 CALIBRATION PROCEDURES

6.1 Field Instruments

Calibration of field instruments will generally be done in the climate-controlled setting of a field office or warehouse/staging area. The calibration procedure will be planned to minimize difficulties and inaccuracies that could result from working in extreme weather and temperature conditions. Calibration procedures for individual instruments that will be used during the field activity are described in the following sections.

The instruments planned for use in the field generally are rugged and designed for use during normal outside weather and temperature conditions. The instruments will be properly cared for and maintained per the manufacturers' instructions. When not in use, the instruments will be stored in the field laboratory trailer or other protected vehicles. During transport from site to site, the instruments will be secured, padded, and protected from jarring, moisture, chemicals, dust, and temperature extremes.

Results of the calibrations will be entered in field notebooks. The field notebooks will be bound, and entries will be made in ink. Notebooks that may be used routinely out-of-doors will be of water-resistant paper, and entries will be made with water-resistant ink. If errors are made in entry, the incorrect information will be crossed out with a single line and the correct information will be entered. No entries will be erased. A separate notebook will be kept for calibration data for each major field activity. The notebooks will be assigned to the leaders of the respective field activities, and each notebook will include title information concerning the name of the person to whom the book is assigned, project and book identification, description of the field activity, date of the field activity, and any other information considered by the field leader to be pertinent to the project. Entries in the field notebooks will be complete and in sufficient detail so that persons unfamiliar with the field activity can understand conditions and results of the operation.

At the completion of the project field operation, the notebooks will be kept in secure storage in the document control center. Persons who are working with the field data will note their custody of the respective notebooks. At the end of the day, the entries in the log books will be signed by the person responsible for that activity.

6.1.2 Photoionization Detector

When used, the photoionization detector (PID) will be calibrated with the natural atmosphere (considered 0 milligrams per liter) and isobutylene gas (100 milligrams per liter) every field day prior to use. Manufacturer calibration instructions will be kept in the field laboratory with the PID. A backup PID will be kept onsite and will be calibrated prior to use.

6.1.3 Thermometer

Before each use, field thermometers will be inspected for cracks, separation of the column, or other defects. The thermometers will be sent to the analytical laboratory and compared to a NITS-certified thermometer in an ice bath and at room temperature to bracket the expected field temperature range. A field thermometer will be rejected if the difference between the two thermometers exceeds 0.5 °C. The rejected thermometers will be disposed of according to manufacturer's specifications. Cooler thermometers will be calibrated using the same procedure, but to 1 °C.

6.1.4 pH Meter

pH meters will be calibrated against two standard pH buffer solutions (4.0 and 7.0, or 7.0 and 10.0) to bracket the expected sample pH. The meter will be calibrated at the beginning of each field day and recalibrated every 4 hours throughout the sampling day. Meters will be checked at the end of the day and any drift in calibration recorded in the field notebook, and measurements adjusted accordingly if the drift exceeds accuracy limits of ±0.2 pH. Buffer solution must be within ±5 °C of sample temperature.

6.1.5 Specific Conductance Meter

The conductivity meter will be calibrated at the beginning of each field day. Specific conductance standards will be used to calibrate the conductivity meters. Conductivity readings will be reported versus a standard temperature of 25 °C.

6.1.6 Dissolved Oxygen Meter

When used, the dissolved oxygen meter will be calibrated in the field twice daily. The probe will be placed in a calibration chamber at ambient water temperature and calibrated for that temperature and pressure, as measured by a field barometer. For measurement of dissolved oxygen, the probe will be placed in flowing water or gently agitated in still water.

6.1.7 Pressure Transducers

When used, pressure transducers will be calibrated before each use. The transducers will be calibrated by placing them in the well and recording readings at measured increments below the water surface.

6.1.8 Geophysical Logging Tools

Geophysical logging tools will be calibrated by the selected logging personnel. The logging engineers will provide documentation as to the frequency and specific techniques of calibration for each probe. The field leader will be responsible for ensuring that the documented procedures and calibration frequencies are followed in the field. Field calibration and QC will be checked by

lowering the geophysical tool in the hole and logging on the way up. The tool will be stopped approximately midpoint of the well and lowered to the bottom again. Logging will then be continued to the surface. This will provide repeat sections to analyze the performance of the tools.

6.1.9 Portable Gas Chromatograph

The portable gas chromatograph will be operated according to manufacturer's instructions. The instrument will be calibrated at the start of operation, midday, and at the end of the work day. Blanks will be included in the calibration. All chromatograms will be saved. The instrument will be calibrated after a steady baseline has been established. Standards will be selected and prepared to target likely potential contaminants and afford an assessment of concentrations of the chemical present in soil gas and serve as a screen to determine if other volatile organic compounds responsive to a photoionization detector are present. Blanks will be prepared from organic-free water (fluid blanks) or from hydrocarbon free (UPC grade) air. The gas chromatograph (GC) will be calibrated by injecting headspace gas from a standard of known concentration. For quantitative screening, incrementally larger aliquots standard will be injected to determine the linearity of the response. Different standard concentrations may be used for calibration, depending on instrument response to the standard selected for use. Throughout the day, the retention time and response of the standards used will be checked. Recalibration will be performed if required.

6.1.10 Current Meters

The pygmy and AA standard current meters are calibrated by towing the meters at a known constant speed (8 different speeds) in a tank of still water. The calibrations are performed in the controlled conditions of a hydraulics laboratory. An average rating is derived for a group of meters, which then becomes the standard rating. The meters are kept in good condition during transport to maintain the standard rating. Prior to each use, a spin test and visual inspection will be made of the meter. If the meter fails the test or inspection, it will be sent to a hydraulics laboratory to be recalibrated.

6.2 Laboratory Equipment

All primary reference standards and standard solutions used by the contract laboratory will be obtained from the NTIS, the EPA Repository or reliable commercial sources. Standards are validated prior to use. Stock and working standards are checked regularly for signs of deterioration, and care is exercised in the proper storage and handling. SOPs for the analytical laboratory for reference standards and solutions is available upon request. Method summaries are listed in Appendix B.

Each instrument is calibrated using manufacturer's methods with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. Quality assurance practices to be followed by the contract laboratory are detailed in Quantera's Quality Assurance Management Plan.

6.2.1 Gas Chromatography/Mass Spectrometry Systems

Each day, prior to analysis of samples, the instrument is tuned with bromofluorobenzene for volatile compounds and decafluorotriphenylphosphine for semivolatile compounds, according to the tuning criteria specified in the applicable SW-846 method. The instrument is then calibrated for all target compounds. An initial calibration curve is produced and certain key compounds are evaluated on a daily basis. If the daily standard does not meet the established criteria, the system is recalibrated.

6.2.2 Gas Chromatography Systems

Initial calibration determines the linear range, establishes limits of detection, and establishes retention time windows. Initial calibration curves consist of five points. Some Appendix IX compounds are calibrated at one point. See Appendix B, table 2 (Method 8080). Acceptable retention time windows for target analytes are ±0.05 minutes or 0.5 percent, whichever is greater. Individual sample matrices can introduce retention time variability and the technical judgement of the analyst is important in making qualitative identification. Calibration check standards are analyzed after every 10 samples. The calibration is checked on a daily basis to ensure that the system remains within specifications. If the daily calibration check does not meet established criteria, the system is recalibrated, and the samples analyzed since the last acceptable calibration check are reanalyzed. For single-component analytes, when target compounds are detected in samples, their presence is confirmed by analysis on a second dissimilar column. For multi-compound analytes, such as arochlors, toxaphene, and chlordane, confirmation is based on comparison of the peak patterns to that of a standard.

6.2.3 Metals Analysis Systems

Each inductively coupled plasma (ICP) emission spectrometer will be calibrated prior to the analyses being performed. The calibration is verified using standards from an independent source. The linear range of the instrument is established once every quarter using a linear range verification check standard. No values are reported above this upper concentration value without dilution. A calibration curve is established daily by analyzing a minimum of two standards, one of which is a calibration blank. The calibration is monitored throughout the day by analyzing a continuing calibration blank and a continuing calibration verification standard. The standard must meet established criteria or the system is recalibrated and all samples analyzed since the last acceptable calibration check are reanalyzed. An interelement check standard is analyzed at the beginning and end of each analytical run, and every 8 hours, to verify that interelement and background correction factors have remained constant. Results outside of the established criteria trigger reanalysis of samples.

Each atomic absorption spectrometer is calibrated prior to analyses being conducted. A calibration curve is prepared with a minimum blank and three standards and then verified with a

standard that has been prepared from an independent source at a concentration near the middle of the calibration range. The calibration is verified on an ongoing basis with a midpoint calibration standard. If the ongoing calibration standard does not meet established acceptance criteria, the system is recalibrated, and all samples analyzed since the last acceptable calibration check are reanalyzed. All samples are spiked to verify the absence of matrix effects or interferences. The sample is diluted or the method of standard additions is used when matrix interferences are present, in accordance with SW-846.

6.2.4 High Pressure Liquid Chromatography System

Initial calibration consists of five points and establishes the linear range of the analysis. Calibration check samples are analyzed every 10 samples. Acceptable limits are 10 percent of the initial calibration. If the calibration check fails, the instrument is recalibrated and all samples analyzed since the last acceptable calibration check are reanalyzed.

6.2.5 Ion Chromatography Systems

Initial calibration consists of establishing the working range of the instrument using a minimum of five standards. Calibration is verified prior to analyzing samples and periodically throughout the run. If the calibration checks are outside of established limits, the system is recalibrated and all samples since the last acceptable calibration check are reanalyzed.

6.2.6 Other

Each system or method is calibrated prior to analysis being conducted. Calibration consists of defining the linear range by use of a series of standard solutions, establishing limits of detection, and identifying potential interferences. The calibration is checked on an ongoing basis to ensure that the system remains within specifications. If the ongoing calibration check does not meet established criteria, the system is recalibrated, and all samples analyzed since the last acceptable calibration check are reanalyzed.

For most methods, a five-point calibration curve is used and a correlation coefficient of 0.995 or greater must be obtained. Calibration checks generally are performed after every 10 samples. The number of points, frequency of calibration checks, and the acceptance criteria depend on the method or system being used and are chosen in accordance to applicable EPA methods. Calibration procedures for each method are summarized in Appendix A. Not all Appendix IX analytes have a five-point calibration curve. For certain compounds, a single low-point calibration standard is analyzed. Then, if these compounds are detected in any of the samples, a five-point curve is generated and the samples are requantitated. Not every Appendix IX analyte is included in the continuing calibration. A shorter list of compounds is included, sufficient to establish that the analytical system is in control.

Additional method summaries are included in Appendix B.

7.0 ANALYTICAL PROCEDURES

7.1 Introduction

The EPA Administrative Order on Consent for Fort Chaffee (3008h) Attachment One, Corrective Action Plan specifies that soil and water analyses for the RFI should be directed at determination of concentrations of 40 CFR Part 264, Appendix IX constituents. In general, analyses will be directed at Appendix IX parameters. However, where a need is determined by the investigators as dictated by conditions at individual sites, additional constituents may be analyzed. All routine analytical procedures will be performed according to SOP as described in SW-846 and 40 CFR, Part 136 (U.S. Environmental Protection Agency, 1986a, 1986b).

The detection limit is the lowest concentration of an analyte that can be detected at a specific confidence level. Definitions of, and procedures for, determining detection limits are presented here.

7.2 Instrument Detection Limit

The Instrument Detection Limit (IDL) is the smallest signal above background noise that an instrument can reliably detect. It is obtained from the analysis of seven replicate standards on 3 nonconsecutive days. The IDL is defined as three times the standard deviation obtained. It should be noted that the IDL does not include the influence of the sample preparation process.

7.3 Method Detection Limit

The Method Detection Limit (MDL) is the minimum concentration of a substance that can be identified, measured, and reported with 99 percent confidence that the analyte concentration is greater than zero. It is obtained by analyzing a minimum of seven replicates spiked at one to five times the expected detection limit. It is calculated by multiplying the standard deviation by the Student t-value for the 99 percent confidence level and number of degrees of freedom (e.g. 3.14 for seven replicates). The procedure is detailed in 40 CFR 136.

The MDL is calculated by multiplying the standard deviation times the Student t-value at the desired confidence level. MDLs for ICP methods, cold vapor atomic absorption method, and colorimetric methods are determined according to procedures outlined in the latest EPA contract laboratory statement of work. The MDL for a routine method is determined by using a reagent water matrix. Method detection limit studies for organic contaminants in soil samples are performed on Ottawa sand.

7.4 Analytical Methods

Specific methodologies along with reporting limits (RLs) are given in Appendix C for all analyses except the acute toxicity biomonitoring (U.S. Environmental Protection Agency, 1985,

1990) and the rapid bioassessment protocols (U.S. Environmental Protection Agency, 1989). The biological counterparts to RLs are based on the individual tests and will be described further in the site work plans.

7.5 Reporting Limit/Project Detection Limit

The reporting limit represents the value above which it is believed reliable data can be obtained on a routine, on-going basis. Reporting limits are established by collecting MDL data for organic analyses and IDL data for metals analyses. These data were compared to limits in EPA publications and regulations. The RL for each analyte is established considering all this information. Using this procedure, the RLs established are generally between two to five times the laboratory MDL/IDL. For the purposes of this study, the RL is synonymous with the project detection limit (Appendix C)

Reporting limits are based on historical recovery data and will be updated periodically. The contract lab will use the most current reporting limits in place at the time of analysis. Current reporting limits at the time of development of this QUAPP are listed in Appendix B. Reporting limits will be provided in the analytical results report for each sample. When changes in analytical methods require the generation of new reporting limits, approved EPA analytical method default limits will be used until historical data are available.

7.6 Tentatively Identified Compounds

Tentatively identified compounds (TICs) are quantified using the total ion current area count for the unknown divided by the area for the nearest internal standard. The amount is calculated assuming a response factor of 1.00 for the unknown and is considered an estimated concentration. Qualitative identification takes place by comparing the unknown compound spectra to the top three library search spectra. The most recent mass spectral library (NITS/EPA/Mass Spectrometry Data Center) is used. Confidence levels will be provided.

8.0 DATA REDUCTION, VALIDATION, AND REPORTING

8.1 Field Data Reduction

Field data reduction will vary depending on the type of data being collected. Data reduction generally will consist of particular analytical procedures that are applied to the data and of the consolidation of data from several sources. Field data will be entered in field notebooks (as described in section 6).

All field data will be entered into a computerized database, which will be a set of relational files that will be grouped by field activities. The database will be able to group, sort, and export data into a wide array of formats. Because the entire set of data will be stored on magnetic media, any desired report format can be output. This will include tabular listings of data, data sorted for selected parameters, and input files for analysis and graphics software. Backups will be made of the entire set of files on a daily basis. Access to the software will be controlled by limiting access to the computer directory where it is stored. Data entry will be validated by storing the key-punched data in a temporary file. A printout of the data in this file will be made and cross-checked by the person entering the data with the original data collection form.

8.1.1 Lithology

Lithologic data from field notes of the drilling and well construction program will be converted into descriptive boring logs. These logs will include data from other sources, such as geotechnical testing. Geophysical logs will be used to assist in identifying transition zones, geologic materials, and for correlation between boreholes.

8.1.2 Hydrology

Hydrologic data may include data from aquifer testing, ground- and surface-water chemistry analysis, water levels from monitor wells, stream discharge data, and acute toxicity biomonitoring (U.S. Environmental Protection Agency, 1985, 1990). Data reduction will include entering field measurements and laboratory analytical results into the computerized data base and analyzing drawdown data from aquifer tests.

8.1.3 Soil

Soil data will consist of laboratory analysis of soil sample chemistry and geotechnical properties. Particular chemical analyses, along with the method of analysis, will be specified in the work plans. Data will be entered into the computerized database when received from the laboratory.

8.1.4 Geophysical Data

Data reduction of geophysical logs will vary depending on the type of log. Logs will be provided in both analog and digital format to facilitate analysis. Surface geophysical surveys will follow a similar format for data reduction.

8.1.5 Ecology

Any ecological data collected will consist of qualitative and quantitative surveys to characterize the terrestrial and aquatic communities, rapid bioassessment monitoring, and sampling of plant and animal tissue at selected sites as specified in the site investigation and work plans. Data reduction will include entering sample data in the computerized data base.

8.2 Laboratory Data Reduction

Raw data resulting from analytical procedures and the equations used by the analyst to calculate reported concentrations are contained in the appropriate analytical reference. In general, EPA guidance will be used. Data reports will be provided by the analytical laboratory by printed copy and on magnetic media to facilitate entry into the database (described in section 8.1).

The analytical laboratory for this RFI study (Quanterra Environmental Services) maintains records of sufficient quality to recreate each analytical event conducted. Details of analytical and QC protocols are contained in Standard Operating Procedures (SPO's). SOP's are documents that contain detailed information on the requirements for the current performance of a laboratory procedure. The laboratory has four categories of SOP's:

SOP's for Performance of an Analytical Method,

SOP's for Preparation of Standards and Reagents,

SOP's for Equipment Operation, Calibration, and Maintenance, and

SOP's for General Laboratory Procedures.

The laboratory relies on a customized Laboratory Information Management System (LIMS) as the primary data base. Client information, sample results, and QC results are all stored in the LIMS. Reports are generated directly from the database to eliminate transcription errors. The most recent 2 years of analytical data are kept on line. All other data are archived on magnetic tape or optical disk.

Laboratory bench sheets and instrument printouts are used to document information from routine laboratory operations, including sample preparation and analysis. Bench sheets are used to ensure that the information is recorded in a complete and organized manner and that the analysis can be reconstructed, if necessary. Portions of information from the bench sheet also are stored in the LIMS.

Laboratory notebooks are used to document information that cannot easily be recorded in the LIMS. Information typically recorded in laboratory notebooks includes unusual observations or occurrences in the analysis of samples, or methods development information. Each page in a laboratory notebook is initialed and dated as information is entered.

A project file is created for each project handled within the laboratory. The project file contains all documents associated with the project. This includes correspondence from the client, chain-of-custody records, raw data, copies of laboratory notebook entries pertaining to the project, and a copy of the final report. When a project is complete, all records are passed to the Document Custodian who inventories the file, checks for completeness, and puts the file into document archive.

Hard copies of field data and notes as well as laboratory test results can be submitted with monthly project status reports if needed. All data will be stored in computer files in accordance with Fort Chaffee guidelines provided to the USGS or using USGS guidelines. All laboratory test data will be included in required tables within the ITR and final reports.

8.3 Field Data Validation

The purpose of data validation is to discover errors, determine sources of errors, and develop approaches to reduce errors. Qualitative validation will generally consist of reviewing how well documented collection procedures were followed and checking the performance of field instruments. Data also will be compared to historical data. In general, the data summaries and reports will be reviewed for transcriptional and typographical errors. Sampling protocols will be reviewed to determine if they were appropriate. The following steps and procedures will be used to ensure that field data will be valid:

- Define data requirement (in site investigation and RFI work plans).
- Define sampling methods (in work plans and FSP).
- Properly calibrate equipment and document calibration.
- Mark field location of sample sites.
- Take sample in accordance with approved procedures.
- Identify sample.
- Take and identify appropriate QA/QC samples.
- Document sample and QA/QC sample collection.

•Take appropriate field measurements.

•Transport samples to appropriate analytical laboratory.

8.3.1 Lithology

Field notebooks will be reviewed for discrepancies between the consolidated boring logs and field drilling notes.

8.3.2 Hydrology and Water Quality

Water chemistry data will be validated by comparing the results of the laboratory analysis of QA/QC samples (section 8.4.1). Data validation techniques for rapid bioassessment will follow EPA (U.S. Environmental Protection Agency, 1985) recommendations. Discharge measurements initially will be verified by visual inspection. If a discharge measurement seems to be questionable, a second check measurement will be made. Water-level data will be field validated by making two measurements that agree within 0.02 foot. Data validation for acute toxicity biomonitoring (where needed) will consist of following the standard procedures outlined in U.S. Environmental Protection Agency, 1990.

8.3.3 Soil

Soil chemistry data will be evaluated through the use of QA/QC samples collected in the field. Geotechnical data will be validated by comparing the raw data to the reported values.

8.3.4 Geophysical Data

Geophysical logs will be compared to drilling logs and repeat sections for qualitative evaluation. Other geophysical measurements will be validated by comparing the raw data to the reported values.

8.3.5 Ecology

Ecological data will be compared with past data and also with data from similar areas (for example, small streams). The rapid bioassessment protocols (U.S. Environmental Protection Agency, 1989) will be used for analysis and validation of macroinvertebrate data.

8.4 Laboratory Data Review and Validation Procedures

Before data can be released from the laboratory, detailed inspection of the results must be conducted. The purpose is to review the data and determine if the data are valid or unacceptable, and to report the data appropriately. Acceptance criteria and corrective actions for each analytical method are listed in Appendix A.

8.4.1 Criteria

The laboratory analyst generating the data has the prime responsibility for the correctness and completeness of the data. Each analyst reviews a data package to ensure that:

•Sample preparation information is correct and complete,

•Analysis information is correct and complete,

•The appropriate SOPs have been followed,

•Analytical results are correct and complete,

•QC samples are within established control limits,

•Special sample preparation and analytical requirements were met, and

•Documentation is complete (all anomalies and holding times have been reported).

The data reduction and validation steps are documented, signed, and dated by the analyst. This initial review step, performed by the analyst, is designated as the level 1 review. The analyst then passes the data package to another reviewer who performs a level 2 review.

The level 2 review is performed by a data review specialist or supervisor whose function is to provide a second review of the data package. This review is conducted according to an established set of guidelines and is structured to ensure that:

•Calibration data are scientifically sound, appropriate, and completely documented,

•QC samples are within established guidelines,

•Qualitative identification of sample components is correct,

•Quantitative results are correct,

•Documentation is complete and correct,

•The data are ready for incorporation into the final report, and

•The data package is complete and ready for data archive.

The level 2 review is structured so that all calibration data and QC sample results are reviewed and all analytical results from 10 percent of the samples are checked back to the benchsheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10 percent of the samples are checked to the benchsheet. The process continues until no errors are found or until the data package has been reviewed in its entirety. All deficiencies will be fully noted, documented, and reported.

An important element of the level 2 review is the documentation of any errors that have been identified and corrected during the review process. The data package submitted by the analyst for level 2 review should be free of errors. Any errors that are found are documented and transmitted to the appropriate supervisor. The cause of each error is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

The level 2 data review is documented and the signature of the reviewer and the date of review recorded. The reviewed data are approved for release and a final report is prepared.

Before the report is released to the U.S. Army, the project data manager will review the report to ensure that the data meet the overall objectives of the U.S. Army. This review is labeled as the level 3 review.

The data manager or project QA officer will review a random 30 percent of the data prior to entry into a computer database to ensure that standard procedures were followed, all QA/QC checks were performed, anomalies were documented, and data packages are complete. The following data validation tasks will be performed on all of the soil and water chemistry data from the analytical laboratory.

- Compare the actual reporting limit to the most current reporting limit,
- Compare sample holding times before preparation to holding time limits required,
- Compare sample holding times before analysis to holding time limits required,
- Evaluate accuracy as percent recovery from matrix spikes,
- Evaluate precision of replicate samples as relative percent difference,
- Evaluate precision of matrix duplicate samples as relative percent difference,
- Evaluate contamination in trip blanks,
- Evaluate contamination in ambient conditions blanks,
- Evaluate contamination in equipment blanks,
- Evaluate contamination in method blanks, and
- Report any compounds detected at a concentration less than the reporting limit, but greater than the IDL.

Copies of all raw data from the analytical laboratory will be made available to the U.S. Army, ADPCE, or EPA upon request to the project chief.

The following items will be evaluated for a random 30 percent of the data (when applicable) to ensure compliance with the QC criteria described in the methodology or in Appendix E.

Volatile Organic Compounds (VOCs) and Semivolatile Organic Compounds-Base/Neutral Acid Extractables (BNAs)

- Gas chromatograph/mass spectrometer instrument tune
- Initial calibration
- Continuing calibration
- Surrogate spikes
- Internal standards
- TICs
- System performance

Inorganics

- Calibration
- ICP interference check samples
- LCSs
- Furnace atomic absorption QC
- ICP serial dilution
- Sample result verification

Pesticides/Polychlorinated Biphenyls

- Gas chromatograph/electron capture detector instrument performance check
- Initial calibration
- Continuing calibration
- Surrogate spikes
- Pesticide clean-up checks

8.4.2 Procedures for Handling Unacceptable Data

When internal laboratory QC finds that results are unacceptable, the source of the problem is determined--standards, instrumentation, or sample preparation. Instrument operational settings,

sensitivity, and linearity are validated. If a problem is detected, it is corrected, and QC samples are reanalyzed. If QC samples are still out of control, then the analytical standards are checked. If the standards are the source of the problem, new or different standards are used and QC samples reanalyzed. If QC data are still outside of limits, the sample preparation is checked for anomalies. If no anomalies are found, the samples are reprepared. If samples cannot be reprepared, the samples are analyzed and the data reported with qualifying information. If sufficient sample remains but holding times have expired, the sample is reprepared and analyzed, and the data are reported with qualifying information. If preparation problems apply only to the QC samples, the samples are analyzed and the data reported with no qualifers. If preparation problems apply only to the QC samples, the samples are analyzed and the data reported with no qualifiers. If preparation problems could have potentially affected all samples, then all samples are reprepared and analyzed. The entire process is documented, including reasons supporting the final decision. Data generated under the circumstances mentioned previously are flagged with an appropriate explanation.

The QA officer will have responsibility for data validation. If any data are found to be unacceptable after review by the data manager and QA officer, they will be noted in the field notebook and on the laboratory data report along with the reasons for unacceptability. These data will not be included in permanent data files, but will be available in hard copy upon request. If otherwise unacceptable data are deemed usable for some reason, the QA officer will provide a narrative supporting the decision.

8.5 Data Validation in Report

All reports will undergo the following validation procedures. Each table or presentation of data will be compared with its original source. Data used in the report will have been validated previously using the procedures described in this QUAPP. Any nonvalidated data that are presented in a report will be qualified. All calculations, technical assumptions, and conclusions will be evaluated by professional colleague review. Report authors will respond to any changes required by the previously defined checks. After corrections are made, a clean copy is printed and compared with the edited copy. This comparison is made by the authors and independently by an editor. Standard operating procedures for tentatively identified compounds are included in section 7.6.

9.0 Internal Quality Control

9.1 Use of Quality Control Samples

QC samples will be used to measure the quality and acceptability of the laboratory data. QC samples collected in the field, such as trip blanks, duplicates, and field equipment blanks, will measure the entire system. QC samples routinely used by the laboratory, such as laboratory control samples, will verify the accuracy and precision of the analytical method and procedures. QC samples will aid in evaluating matrix effects on the various analytical methods and in assessing the suitability of the sampling procedures. Appendix A provides a summary table of internal quality control procedures.

9.2 Field Quality Control Samples

Environmental samples are the actual samples used to quantify contamination. QC samples will supplement environmental samples. QC samples will be collected in the field for both soil chemistry and water chemistry. The frequency and timing of collection for the various QC sample types are defined in the FSP.

9.2.1 Soil Chemistry Samples

Soil chemistry QC samples will consist of field replicates, samples for matrix spiking, and equipment blanks.

Field Replicates for Soil Sampling

Field replicate sample analysis will measure the precision of the entire system. Replicates will be collected from the same sample core barrel as the primary sample. The sample replicates will be marked with a fictitious designation that will be logged in the field notebook. They will be identified as a replicate in the field notebook. After analysis, the sample will be identified as a replicate sample in the computer data base.

Matrix Spikes and Matrix Spike Duplicates for Soil Sampling

The sample sets selected for matrix spike and matrix spike duplicates (MS/MSD) analysis will contain a primary soil sample and samples for duplicate spiked analysis. Samples for MS/MSD analysis will measure the effects of the sample matrix and provide an estimate of accuracy. These samples will all be taken from the same core barrel. Each sample is collected from the core barrel sample length, generally 5 feet or less. Compounds and concentrations of spiking standards are given in Appendix B.

Equipment Blanks for Soil Sampling

Equipment blanks will be taken as a check of decontamination procedures and to ensure that new equipment is not introducing contamination into the system. Equipment blanks for soil sam-

ples will be prepared by rinsing a decontaminated soil sampler with deionized water and collecting this rinse water. The frequency of equipment blanks will be specified in the FSP. A soil sample site is defined as one borehole (soil boring, well, or test hole) where a series of soil samples may be taken.

9.2.2 Ground-Water Samples

Ground-water QC samples will consist of trip blanks, ambient condition blanks, field replicates, samples for matrix spiking, and equipment blanks.

Trip Blanks for Ground-Water Sampling

Trip blanks will be collected to detect contamination of volatile organic compound samples potentially introduced from sample containers and during transportation to and from the laboratory. A trip blank consists of a set of sample bottles filled with reagent-grade water in the laboratory. This set of sample containers will accompany the empty sample bottles to the site. During sampling, the bottles will remain closed. They will be packaged and sent to the laboratory for analysis with the environmental samples. The appropriate container for each analysis will be the same as the environmental samples for the constituents of interest.

Ambient Conditions Blanks for Ground-Water Sampling

Ambient conditions blanks serve a similar purpose as trip blanks with the exception that they do not measure the entire system. Ambient conditions blanks are exposed to the field environment (air) to detect any contamination that may be introduced while collecting samples. They are collected for VOCs and will assess ambient conditions at the sampling site and in the sample coolers. Ambient conditions blanks are filled with reagent grade water at the sampling site and treated as regular samples.

Field Replicates for Ground-Water Sampling

Field replicate sample analysis will measure the precision of the entire system. Replicates will be collected during the same period of pumpage as the primary sample. The sample replicate will be marked with a fictitious designation which will be logged in the field notebook. The real and fictitious identities will be documented in the field notebook.

Matrix Spikes and Matrix Spike Duplicates for Ground-Water Sampling

MS/MSDs for water samples serve the same purpose as for soil sampling. The sample sets selected for MS analysis will contain a primary water sample and samples for MS and MSD analysis. These samples will be collected during the same period of pumpage as the primary sample. Compounds and concentrations of spiking standards are given in Appendix B.

Equipment Blanks for Ground-Water Sampling

Equipment blanks will be taken as a check of decontamination procedures and to ensure that equipment is not introducing contamination into the system. Equipment blanks will be prepared by filling a pump-decontamination stand pipe with reagent-grade water and pumping the water through the pump intake line, the sampling pump, and the discharge line for collection. All equipment will be decontaminated prior to the procedure. Equipment blanks will be collected for each analysis that is being performed at a site chosen for equipment blank sampling. The frequency of equipment blanks will be specified in the FSP.

9.2.3 Surface-Water Samples

Surface-water samples may be augmented by trip blanks, ambient conditions blanks, field replicates, matrix spikes, and matrix spike duplicates. Equipment blanks generally will not be required since the sample bottles will be filled directly from the source. Surface-water QC samples serve the same purpose as ground-water QC samples.

Trip Blanks for Surface-Water Sampling

The procedures for trip blanks will be the same as described in section 9.2.2.

Ambient Conditions Blanks for Surface-Water Sampling

Ambient conditions blanks will be collected following the sample procedures in section 9.2.2.

Field Replicates for Surface-Water Sampling

Field replicate sample analysis will measure the precision of the entire system. Replicates will be collected from the same area as the primary sample. The sample replicates will be marked with a fictitious designation that will be logged in the field notebook.

Matrix Spikes and Matrix Spike Duplicates for Surface-Water Sampling

The sample sets selected for MSD analysis will contain a primary sample and samples for duplicate spike analyses. These samples will be taken from the same area. Compounds and concentrations of spiking standards are given in Appendix B.

9.2.4 Wipe Sample Quality Assurance/Quality Control Samples

Background Sample (referred to as field blank in EPA PCB Spill Cleanup Policy, 1987)- at least 5 percent of the total samples include at least two samples each from the following:

a. Ship unopened vials back to lab for analysis.

b. With gloved hands, remove the cap from a sample vial for the estimated time (record this time) of normal wipe sampling, allow the gauze to air dry without applying it to any surface, and proceed to insert wipe in vial and cap tightly.

c. Use the wipe sampling procedures to wipe some areas/surfaces near the sampling site that are not expected to be contaminated.

Duplicates - at least 5 percent of total samples including at a minimum the designated samples from the following groups:

a. Double wipe at least two sample sites, label which was the first wipe and which was the second wipe for each of the two sites, for each type of surface sampled.

b. For at least two sample sites, for each type of surface sampled, wipe two adjacent identical areas. Clearly identify the samples as being adjacent to one another in the sample description forms.

Field Spikes - at least 5 percent of total samples including at a minimum the designated samples from each type of surface sampled. Clearly describe the samples on the description forms.

a. For two vials or more, remove each gauze and moisten as for sampling and spike each wet gauze with 10 µg each of the type of PCB's or other analytes that were spilled, wipe a contaminated surface adjacent to a sampled surface as in Duplicates-b above, let the gauze air dry, replace the gauze, and proceed to insert wipe in vial and cap tightly.

b. For a second pair of vials or more, remove each gauze and moisten as for sampling, wipe a contaminated surface adjacent to a sampling surface as in Duplicates-b above, after wipe sampling (but before air drying), spike each wet gauze with 10 µg each of the type of PCB's or other analytes that were spilled, let the gauze air dry, replace the gauze in the vial and tighten cap.

c. For a second pair of vials or more, spike the sampling surfaces adjacent to another sampled surface as in Duplicates-b above with 10 µg each of the types of PCB's or other analytes that were spilled and allow to air dry. Remove each gauze and moisten as for sampling; wipe the surface; let the gauze air dry, replace the gauze in the vials and proceed to place in vial and tighten cap.

9.3 Laboratory Quality Assurance/Quality Control Samples

The contract laboratory QA/QC program consists of the operational controls used to ensure that data generated meet predefined requirements of precision and accuracy, and that the system instituted documents the effectiveness of these controls: The QA/QC program has provisions to meet the following two objectives: (1) to monitor the laboratory's daily performance of an analyt-

lytical method. Laboratory QA/QC is defined in the Quality Assurance Program Plan for Environmental Chemical Analysis.

9.3.1 Laboratory Control Samples (LCSs)

LCSs are well-characterized, laboratory-generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCSs for aqueous samples consist of deionized or carbon-filtered laboratory reagent water spiked with a group of target analytes selected to represent the specific method being used. The matrix for soil sample LCSs is standard Ottawa sand for organics only. LCSs are analyzed in duplicate with each lot of 20 samples, or, if the analytical lot is smaller, with each analytical lot.

Three types of LCSs are routinely analyzed: duplicate control samples (DCSs), single control samples (SCSs), and method blanks.

Duplicate Control Samples

DCSs are used to monitor the precision and accuracy of the analytical system on an ongoing basis. DCS consist of standard, control matrices that are spiked with a group of target compounds representative of the method analytes. A DCS pair is analyzed for every 20 samples processed by the method.

Single Control Sample

Samples often are analyzed in lots of less than 20 because of holding time or turn-around time requirements. SCSs provide a measure of laboratory performance with each batch of samples processed. A SCS consists of a control matrix that is spiked with surrogate compounds appropriate to the method being used. In cases where no surrogate is available (metals or conventional analyses), a single DCS serves as the control sample. A SCS is prepared for each sample lot for which the DCS pair are not analyzed. Recovery data generated from the SCSs are compared to control limits that have been established for each of the compounds being monitored.

Method Blanks

Method blanks are analyzed to assess the level of background interference or contamination that exists in the analytical system and that might lead to the reporting of elevated concentration levels or false positive data. A method blank is analyzed with every batch of samples processed. A method blank consists of reagents specific to the method that are carried through every aspect of the procedure, including preparation, clean-up, and analysis. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples.

9.3.2 Surrogate Recovery Samples

Surrogate recovery samples (SRSs) are used for all organic analyses. An SRS consists of blanks, samples, and standards. Surrogate compounds are added to all blanks, all samples, and all standards. Surrogates are compounds that are chemically similar to the analytes of interest, expected to behave similar to the analytes of interest, and not expected to be found in environmental samples. A SRS is prepared with each lot of samples.

9.3.3 Matrix Spikes

A MS is a sample to which known concentrations of analytes have been added. The MS is taken through the entire analytical procedure, and the recovery of the analytes is calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of the sample matrix on the accuracy of the analysis.

9.3.4 Matrix Spike Duplicates

A MSD is a sample that is divided into two separate aliquots, each of which is spiked with known concentrations of analytes. The two spiked aliquots are processed separately and the results compared to determine the effects of the matrix on the precision and accuracy of the analysis. Results are expressed as RPD and percent recovery.

9.3.5 Matrix Duplicates

A matrix duplicate is a sample that is divided into two separate aliquots. The aliquots are processed separately and the results compared to determine the effects of the matrix on the precision of the analysis. Results are expressed as RPD.

9.3.6 Control Limits

Control limits for the LCS/SRS programs are taken from the applicable method or, where data are available, are established based on the laboratory's historical data for QC samples. The LCS/SRS programs provide a proactive means of making consistent, accurate decisions regarding the quality of a particular set of data. Data that are generated with a SRS or LCS that falls within acceptance criteria are deemed to be in control. Data that are generated with a SRS or LCS that falls outside of acceptance criteria are considered suspect and must be reanalyzed or reported with qualifiers that explain in a narrative how the data are out of control. The subsequent use of these data will depend on this qualifier.

Laboratory control limits for quality control samples--surrogate, laboratory control samples, and matrix spike samples--recoveries are based on historical recovery data and will be updated periodically as required. The contract laboratory, Quanterra Analytical Laboratories, will use the most current control limits in place at the time of analysis to evaluate laboratory performance.

Current control limits at the time of preparation of this QUAPP are presented in Appendix B. The control limits will be provided in the analytical results report for each sample. Surrogate and spike concentrations may change because of the availability of reference materials or upon client request.

9.3.7 Documentation

Documentation is handled primarily through the laboratory information management system (LIMS). The sample laboratory identification number, other sample information, client information, dates of extraction and analysis, QC results, and sample results are all stored in the LDMS. Reports are generated directly from the database. A tiered security system is in place and a system audit trail identifies when information has been changed and who changed it. A project file is maintained for each project handled within the laboratory. This file contains all documents associated with the project, including any correspondence from the client, chain-of-custody records, raw data, copies of laboratory bench notebook entries pertaining to the project, and a copy of the final report. The file is archived when the project is complete.

10.0 PERFORMANCE AND SYSTEM AUDITS

The project QA officer will evaluate the entire system, both field and laboratory, once data are available. The laboratory analytical data reports will be reviewed for completeness as compared to the initial request. The results of field QC will be evaluated to determine the effectiveness of sample collection techniques. The entire data package for each sample/site will be reviewed as a whole to determine the adequacy of the measurement system to provide data of known quality and quantity to meet the project objectives. This audit will be performed as frequently as necessary, generally after all analytical data have been received for a sampling session at a site or for a suite of similar analyses conducted at a number of sites.

10.1 Field Audits

Oversight of field QA/QC procedures will be the responsibility of the QA/QC manager. He will review all elements of the individual investigation work plans and FSPs to ensure that proper QA/QC procedures are followed and ensure that site investigation and RFI objectives are met.

10.1.1 Field Sampling

Field sampling activities will be reviewed in two primary areas: field sampling procedures, and health and safety procedures. Field techniques to meet criteria for both areas are covered in detail in the FSP and health and safety plan. In addition, each investigation work plan will detail site-specific procedures and techniques. The field leader will maintain QA/QC records of all field activities. This information will be entered into the field notebooks as specified in the FSP.

10.1.2 Documentation

QA/QC procedures and information entered into the field notebooks will be kept as permanent record. The results of reviews by the project QA/QC officer will be reported in memorandum form for the Fort Chaffee Environmental Branch as requested. As an oversight activity to the U.S. Army, field audits may be performed by the EPA and ADPCE. Records of these audits will be kept by the EPA with a summary provided to the USGS and the U.S. Army.

10.2 Laboratory Audits

Laboratory audits are performed as a regular part of the laboratory QA/QC program. External audits are performed on a regular basis by outside agencies. Internal audits are performed by the laboratory to ensure that documented QA/QC procedures are routinely followed.

10.2.1 External

The laboratory participates in external audits to ensure that sample control, analysis, data, and documentation meet stringent regulatory requirements and the procedures comply with good laboratory practices.

10.2.2 Internal

In addition to external audits, internal audits are performed regularly. Monthly system audits are conducted by the laboratory's QA/QC section or officer. Laboratory QC check samples are analyzed at a rate of approximately 10 percent of the total sample workload. Control charts are maintained and monitored daily by QC personnel to determine out-of-control situations or detect trends. Internal audit problems generally are acted upon at the bench level by the analyst or supervisor. Corrective actions are documented.

The laboratory is subjected to quarterly systems audits by the QA department. These audits are intended to serve two purposes: (1) to ensure that the laboratory is complying with the procedures defined in laboratory manuals and the QUAPP, and (2) to determine any sample flow or analytical problems. The frequency of the audits will be increased if any problems are suspected. A corporate QA audit will be performed on an annual basis by the corporate director of quality assurance. This audit is intended to check compliance with the laboratories' overall QA program. All audits by divisional and corporate QA staff are performed more frequently, or specifically directed audits are performed if workload increases or if any problems are suspected in the laboratory.

10.2.3 Documentation

The results of both internal and external audits are documented. Internal audit results will be included with the data that were being analyzed at the time of the audit. External audit results will be made available to the U.S. Army project manager and the RI site manager. Upon U.S. Army approval, results of these audits will be released to EPA representatives.

11.0 PREVENTIVE MAINTENANCE

11.1 Field Equipment

Field equipment will be properly calibrated, charged (as specified by the manufacturer), and in good general working condition prior to the beginning of each working day. All field instruments will be protected against inclement weather during the field investigations. Field equipment will be maintained according to the manufacturers instructions and will arrive at the site each day in proper working condition. All lubrication, hydraulic oil, and motor oil will be checked to make certain fluid reservoirs are full and there are no leaks. Batteries, spare pH electrodes, and spare instruments and equipment will be available onsite and are the responsibility of the field leader. Current meters will be checked after each measurement to determine if the rotor is moving freely and that it doesn't stop abruptly. The current meter will be cleaned and inspected thoroughly to make sure all components are in workable condition. The meters will be tested by spinning the rotor and timing how long it takes the rotor to come to a complete stop. The pygmy meter must spin a minimum of 45 seconds while the AA standard meter must spin a minimum of 2 minutes. Field instruments will be serviced and overhauled by the manufacturer as needed and a log of these services maintained in the District office of USGS.

11.2 Laboratory Equipment

Preventive maintenance is performed routinely by designated laboratory personnel on a schedule specific for each system. When repairs are necessary, they are performed by either specially trained staff or trained service engineers employed by the instrument manufacturer. Arrangements are made with the various instrument manufacturer's representatives to keep spare parts on hand, and in some cases, entire backup analytical systems. The laboratory is large enough to have a substantial number of identical or nearly identical analytical systems online. If one system fails, another is immediately available to serve as a backup until repairs can be made.

12.0 ROUTINE PROCEDURES TO ASSESS DATA

12.1 Precision, Accuracy, and Completeness

The specific routine procedures used to assess data are precision, accuracy, and completeness. General definitions of these parameters are given in section 3 of this document. Specific formulas are given here.

12.1.1 Precision

Precision of field measurements will be ensured by taking multiple measurements of a single property. Precision of field data will be measured by comparing the results of the environmental samples with the field replicate samples. The precision of laboratory analyses will be assessed by comparing the analytical results between MS/MSD samples for organic analyses, and laboratory duplicate analyses for inorganic analyses. The RPD will be calculated for each pair of duplicate analyses as follows:

$$RPD = \frac{|D1 - D2|}{(D1 + D2)/2} \times 100\%$$

where,

RPD is relative percent difference

D1 is first sample value, and

D2 is second sample value.

12.1.2 Accuracy

Accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy of laboratory results will be assessed using the analytical results of surrogate spikes, LCSs, and matrix spikes. Accuracy may be calculated in terms of percent recovery as follows:

$$PercentRecovery = \left(\frac{S - U}{T}\right) \times 100\%$$

where,

S is the result for the spiked sample,

U is the result for the unspiked sample, and

T is the amount spiked.

12.1.3 Completeness

The data will be assessed for compliance with the amount of data required for decision making. The percent completeness for each data set is calculated as follows:

$$C = \frac{DO}{DP} \times 100\%$$

where,

C is completeness,

DO is data obtained, and

DP is data planned.

12.2.1 Record Review

Field records and analytical data will be reviewed to verify data accuracy and validity and to ensure that the environmental conditions interpreted for a particular site are technically sound. Data generated under the laboratory and field QA/QC program will be used to evaluate the analytical results assembled for each site and to formulate conclusions and recommendations pertaining to the need for additional site investigations. This data quality assessment will be presented in the appropriate sections of the Technical Report for the RFI.

Field records will be evaluated for completeness to ensure that the requirements in the Work Plan have been fulfilled and the procedures documented in the SAP have been implemented. Anomalous field data will be identified and explained to the extent possible.

A review of laboratory data completeness will be done to ensure that all samples and analyses required by the SOW have been processed, complete records exist for each analysis and the associated QC samples, and that the procedures specified in the Work Plan and SAP have been implemented. Analytical control and detection limits will be assessed with any control limits outside of the acceptable range as specified in the SAP identified along with any trends or problems with the data. Any detection limits that exceed the limits in the SAP will also be identified. Sample-holding times will be compared with limits described in the SAP. If holding times are exceeded, the site will be resampled.

Precise field duplicate results indicate reproducible sampling technique and precise laboratory analysis. Field duplicate results not within control limits could indicate a heterogeneous sample medium, poor sampling technique, or a lack of analytical precision. Results from the analysis of blanks will be assessed to determine sources of contamination and the impact of any contamination on the analytical results for environmental samples. Potential matrix effects identified through laboratory tests or field records will be identified and their impact on results for environmental samples shall be described in the technical report.

Data that are not reasonable representatives of environmental conditions will not be used in the evaluation process. This determination will be made using the professional judgment of a multidisciplinary team of geologists, hydrologists, chemists, and other personnel having direct experience with the data-collection effort. This coordination is essential for the identification of valid data and the proper evaluation of that data.

13.0 CORRECTIVE ACTIONS

13.1 Out-of-Control Events

13.1.1 Field Activities

Out-of-control events in the field occur when certain conditions prevent the use of standard or prescribed techniques, and alternate or nonstandard techniques/procedures must be used. The reason for the difficulty or problem must be identified and the field lead notified. Corrective action documentation will be in the appropriate, bound, field notebook.

13.1.2 Laboratory Activities

Laboratory analytical data are considered out of control when:

- QC data outside the warning or acceptable windows for precision and accuracy,
- Blanks or control samples contain contaminants above acceptable levels,
- Analyses are out of control for more than one batch of samples,
- Changes in detection levels occur,
- Deficiencies are detected during internal or external audits or from the results of performance evaluation samples, or
- Inquiries concerning data quality are received from clients.

Responses to an out-of-control event in the laboratory are as described in section 8.4. Initial responsibility for declaration, notification, and documentation rests with the analyst. Any person involved in the laboratory data review also has responsibility for ensuring the proper procedures are followed and documented. Corrective actions related to specific laboratory methods are listed in Appendix A.

13.2 Principal Corrective Action

The principal corrective action that might be required as a result of an out-of-control event would be resampling. An indication of possible corrective action can be noted by any person responsible for collection, reduction, validation, or other data-related duty and should be reported to one of the following project personnel:

- project chief,
- field work team leader, or
- quality assurance officer.

One of these three personnel should review the data in question and initiate corrective action when needed.

14.0 QUALITY ASSURANCE REPORTS

Effective management of field sampling and analytical efforts requires timely assessment and review of activities. This will require effective interaction and feedback between field team members, the analytical laboratory, and the project supervisors. The field team and project supervisors will communicate daily.

14.1 Field Status Reports

A status report will be prepared as requested by the field work team leader for the project chief and the QA/QC officer. The QA/QC officer will review and compile these reports and combine them with any onsite audits conducted into an annual report for the project manager. When laboratory analytical data are received, the laboratory QA reports will be included in the QA report to the project manager. These reports will address the following:

- Summary of activities and general program status,
- Summary of calibration and QC data,
- Summary of unscheduled maintenance activities,
- Summary of corrective action activities,
- Status of any unresolved problems,
- Assessment and summary of data completeness, and
- Summary of any significant QA/QC problems and recommended and implemented solutions not included above.

14.2 Laboratory Status Reports

Laboratory quality assurance reports are prepared monthly by laboratory QA personnel and submitted to laboratory managers. These reports will include:

- Results of the monthly systems audit, including any corrective actions taken,
- Performance evaluation scores and commentaries,
- Results of site visits and audits by regulatory agencies and clients,
- Performance on major contracts,
- Problems encountered and corrective actions taken,
- Holding times violations, and

Comments and recommendations.

In addition, a weekly summary of the internal QA audit for reported data is sent to laboratory QA senior mangers.

15.0 REFERENCES CITED

Occupational Safety and Health Administration, 1989, Hazardous waste operations and emergency response: Occupational Safety and Health Administration, Final Rule, 29 CFR Part 1910.

U.S. Environmental Protection Agency, 1985, Methods for measuring the acute toxicity of effluents to freshwater and marine organisms: Cincinnati, Ohio, EPA/600/4-85/013, 216 p.

----- 1986a, Guidelines establishing test procedures for the analysis of pollutants under the Clean Water Act: U.S. Environmental Protection Agency, 40 CFR Part 136.

----- 1986b, Test methods for evaluating solid waste, 3rd edition, Update II: Office of Solid Waste and Emergency Response, EPA SW-846.

----- 1987, Data quality objectives for remedial response activities, development process: U.S. Environmental Protection, EPA/540/G-87/003.

----- 1988, Guidance for conducting remedial investigations and feasibility studies under CERCLA: Denver, Colorado, U.S. Environmental Protection Agency, EPA/540/G-89/004.

----- 1989, Rapid bioassessment protocols for use in streams and rivers; benthic macroinvertebrates and fish: EPA/444/4-89-001, variable pagination.

----- 1990, Region VIII NPDES whole effluent toxics control program December, 1990: Denver, Colorado, U.S. Environmental Protection Agency, 26 p.

APPENDIX A

SUMMARY OF INTERNAL QUALITY-CONTROL PROCEDURES

Summary of internal quality-control procedures

[h, hour; RPD, relative percent difference; NIST, National Institute of Standards and Technology; ICPAES, inductively coupled plasma atomic emission spectroscopy; PDL, project detection limit; QC, quality control; DCS, duplicate control sample; SD, standard deviation; MS/MSD, matrix spike/matrix spike duplicate; RI, remedial investigation; MEK, methyl ethyl ketone; SCS, surrogate control sample; IS, internal sample; IS, internal standard; PCBs, polychlorinatedbiphenyls; conc., concentration; EPA, Environmental Protection Agency; MeCl2, methylene chloride; USATHAMA, U.S. Army Toxic and Hazardous Materials Agency; ASTM, American Society for Testing Materials; CLP, contract laboratory program; µg, microgram; TOX, total organic halides; mg, milligram; TCLP, toxic characteristic leaking procedure; <, less than; >, greater than; S/N, signal to noise]

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
E150.1	pH, onsite	Calibrate meter	Prior to first use each day and every 2 h.	Each of three different buffer solutions (4,7,10) read within 0.1 pH unit.	Clean or replace electrode or obtain new meter.
		Check meter calibration	Prior to each measurement series at one location	Two buffers (4 and 7; or 7 and 10) bracketing the sample pH read within 0.1 pH unit of initial calibration.	Recalibrate meter using three buffers; if necessary clean or replace electrode or obtain new meter.
A403	Alkalinity, onsite	Standardize titrant	Initially and every 10 samples	Normality of titrant is within 10 percent of laboratory-determined value.	If titrant is contaminated, obtain fresh supply.
		Duplicate	1 duplicate per 10 samples	Within 20 percent RPD.	Reanalyze same duplicate set. Determine if lack of precision is caused by analytical or instrumental problems or is caused by chemical changes occurring in samples. If caused by analytical or instrumental problems, correct problems and reanalyze affected samples. If caused by chemical changes occurring after sample collection, document situation.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
E120.1	Specific conductance, onsite	Calibrate meter	Initially in laboratory at 3 or more points; verified daily at point near value of sample	Within 5 percent of known value of specific conductance standard.	Clean or replace probe, recalibrate.
E170.1	Temperature, onsite	Calibrate thermometer	Initially in laboratory at 2 points against NIST thermometer	Within 0.5 degrees of NIST thermometer.	Obtain new field thermometer.
		Visual inspection	Daily	No column breaks, cracks, chips, or scratches	Obtain new thermometer and calibrate.
SW6010	Metals (ICPAES)	Equipment blank	Specified in RI work plans	Any compounds detected to be less than 3 times the PDL.	If compounds found in equipment blank also are found in samples at similar concentrations, determine cause, evaluate effect on samples, and qualify data or resample if necessary.
		Field replicate	1 set per 10 samples	Within 30 percent RPD for water samples[a] and within 40 percent for soils.	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control
		Calibration curve	Established daily, verified every 10 samples	Initial calibration verification check standard is within 10 percent of calibration curve.	1. Validate standard. If standard still exceeds acceptance criteria, obtain fresh, certified standards. 2. Recalibrate instrument. 3. Document actions taken.
		Interelement check sample	Daily	Concentration of analyte subject to potential spectral interference is within 20 percent of known value.	1. Recalibrate instrument and reanalyze interelement check sample. 2. Check interelement correction factors and update if necessary. 3. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Method blank	1 per batch or 1 per 20 samples	Concentration does not exceed two times the PDL of the analyte.	1. Determine if concentration levels in the sample exceed 10 times concentration levels in blank. If so, qualify data. 2. If not, reprepare blank, QC samples and affected samples. 3. Document actions taken.
		DCS	1 duplicate per 20 samples	80 percent of values are within three SD of mean historical values or methodology control limits of precision and accuracy. Acceptance criteria are in Appendix B[b].	1. Validate instrument parameters, sensitivity, and linearity. Correct problems and document standards. 2. Validate standards. 3. Validate DCS preparation. 4. Reanalyze DCS and samples. 5. If repreparation of samples if not possible, qualify data. 6. Document actions taken.
		MS/MSD	1 per 20 samples	Acceptance criteria are in Appendix B.	1. Verify that the spike concentrations is at least 50 percent of the unspiked concentration. 2. Verify that correct spiking solutions and amounts were used. 3. Check method blanks and DCS recovery. 4. Reanalyze samples if laboratory error is suspected. 5. Document actions taken.
SW7060 SW7421 SW7470 SW7471 SW7740	Arsenic Lead Mercury Mercury Selenium	Equipment blank	Specified in RI work plans	Any compounds detected are less than 3 times the PDL.	If compounds found in equipment blank also are found in samples at similar concentrations, determine cause, evaluate effect on samples, and qualify data or resample if necessary.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Field replicate	1 set per 10 samples	Within 30 percent RPD for water samples[1] and within 40 percent RPD for soils.	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control
		Calibration curve	Established daily, verified every 10 samples	Verification check standard is within 10 percent of calibration curve. Correlation coefficient for mercury equals or exceeds 0.995.	1. Validate standard. If standard still exceeds acceptance criteria, obtain fresh, certified standard. 2. Recalibrate instrument. 3. Document actions taken.
		Method blank	1 per batch or 1 per 20 samples	Concentrations are less than two times the PDL of the analyte.	1. Determine if concentrations in the samples exceed 10 times concentrations in blank. If so, qualify data. 2. If not, reprepare blank, QC samples and affected samples. 3. Document actions taken.
		Analytical spike	Every sample (except mercury) Recovery analysis for mercury is performed with MSs/MSDs	Recovery is within 80-120 percent.	1. If recovery exceeds 120 percent, then standard additions will be performed. 2. If recovery is less than 40 percent or greater than 200 percent, the sample must be diluted and reanalyzed (including an analytical spike on the diluted sample). 3. If the criteria are not met and the sample result is below the reporting limit, report the result as less than twice the PDL. 4. If the criteria are not met and the sample result is greater than the PDL, reanalyze the sample using the method of standard additions. 5. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		DCS	1 duplicate per 20 samples	Values are within three SD of mean historical values or methodology control limits of precision and accuracy. Acceptance criteria in Appendix B.	1. Validate instrument parameters, sensitivity, and linearity. Correct problems and document action taken. 2. Validate standards. 3. Validate DCS preparation. 4. Reanalyze DCS and samples. 5. If repreparation of samples is not possible, qualify data. 6. Document actions taken.
		MS/MSD	1 per 20 samples	Acceptance criteria are in Appendix B.	1. Verify that the spike concentration is at least 50 percent of the unspiked concentration. 2. Verify that correct spiking solutions and amounts were used. 3. Check method blanks and DCS recovery. 4. Reanalyze samples if laboratory error is suspected. 5. Document actions taken.
E300 SW9010 E350.1 E353.2	Sulfate, chloride, fluoride Cyanide, total amenable Nitrogen, ammonia Nitrogen, nitrate	Equipment blank	Specified in RI work plans	Concentration of analyte is less than three times the PDL.	If compounds found in equipment blank also are found in samples at similar concentrations, determine cause, evaluate effect on samples, and qualify data or resample if necessary.
E354.1 E353.2	Nitrogen, nitrite Nitrogen, nitrate + nitrite	Field replicate	1 set per 10 samples	Within 30 percent RPD for water samples[1] and within 40 percent for soils.	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
E351.2 E365.3	Nitrogen, total Kjeldahl Phosphorous, total	Calibration curve	Established daily, verified every 10 samples	Verification check standard is within 10 percent of calibration curve. Correlation coefficient of calibration curve equals or exceeds 0.995.	1. Validate standard. If standard still exceeds acceptance criteria, obtain fresh, certified standard. 2. Recalibrate instrument. 3. Document actions taken.
		Method blank	1 per batch or 1 per 20 samples	Concentration is less than two times the PDL of the analyte.	1. Reprepare all blanks, QC samples, and samples. 2. Document actions taken.
		DCS	1 duplicate per 20 samples	Values are within three SD of mean historical values or methodology control limits of precision and accuracy. Acceptance criteria are in Appendix B[2].	1. Validate instrument parameters, sensitivity, and linearity. Correct problems and document action taken. 2. Validate standards. 3. Validate DCS preparation. 4. Reanalyze DCS and samples. 5. If repreparation of samples is not possible, qualify data. 6. Document actions taken.
		MS/MSD	1 per 20 samples	Acceptance criteria are in Appendix B.	1. Verify that the spike concentration is at least 50 percent of the unspiked concentration. 2. Verify that correct spiking solwtions and amounts were used. 3? Check method blanks and DCS recovery. 4. Reanalyze samples if laboratory error is suspected. 5. Document actions taken.
E160.1	Dissolved solids	Field replicate	1 set per 10 samples	Within 30 percent RPD[1].	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Method blank	1 per batch or 1 per 20 samples	Concentration is less than two times the PDL.	1. Determine and correct problem. 2. Reanalyze batch, including QC samples, if necessary. 3. Document actions taken.
		DCS	1 duplicate per 20 samples	Within 90-110 percent accuracy and 10 percent precision.	1. Determine and correct problem. 2. Reanalyze samples, if necessary. 3. Document actions taken.
SW8080	Organochlorine pesticides and PCBs	Equipment blank	Specified in RI work plans	Target compounds are not present at concentrations greater than the PDL.	If compounds found in equipment blank also are found in samples at similar concentrations, determine cause, evaluate effect on samples, and qualify data or resample if necessary.
		Field replicate	1 set per 10 samples	Within 30 percent RPD[1].	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control.
		Calibration curve	Established at 5 conc. levels for single-peak analytes and at single conc. for multi peak analytes. Verified daily at mid level.	Verification check standard is within control limits established for the EPA contract laboratory program.	1. Reanalyze check standard. 2. If similar results are obtained recalibrate instrument. 3. Reanalyze samples processed since last time criteria were met. 4. Document actions taken.
		Method blank	1 per batch or 1 per 20 samples	Concentration is less than the PDL of the analyte.	1. Determine source of contamination, that is, instrument, blank water, or reagents. 2. Take appropriate corrective action and document actions taken. 3. If preparation is in error, reanalyze or reprepare batch. 4. If samples cannot be reanalyzed or reprepared, qualify data. 5. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		DCS	1 duplicate per 20 samples	Recovery and precision for 80 percent of the analytes are within limits established for matrix spike and matrix spike duplicate shown in Appendix B.	1. Validate instrument parameters, sensitivity, and linearity. Correct problems and document actions taken. 2. Validate standards. 3. Validate DCS preparation. 4. Reprepare and reanalyze DCS and affected samples. 5. If repreparation of samples is not possible, qualify data. 6. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Surrogate spike	All blanks and samples	Surrogate spiking compounds, spike concentrations, and control limits are in Appendix B.	1. Examine all QC (including but not limited to DCS, SCS, internal standards, and instrument check standards). 2. If surrogate in SCS is out of control, check quantification. If quantification is acceptable, reanalyze SCS. 3. If similar results are obtained from reanalysis, and both the SCS and surrogate in the samples are similarly affected, obtain fresh, certified SCS solution and reprepare QC samples and all affected samples. 4. If only the SCS and not the samples are affected, report data and qualify. 5. If samples also are affected but cannot be reprepared, qualify data. 6. If surrogate spike in SCS is acceptable but out of control for samples, validate preparation of samples. If no error or problems are discovered for sample preparation, qualify data. 7. If errors are discovered in preparation of samples, reprepare QC samples and all affected samples. 8. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		MS/MSD	1 set per 20 samples (Collector to provide duplicate samples)	Matrix spiking compounds, spike concentrations, and control limits are in Appendix B.	1. Analyze spiking solution. 2. If spiking solution is valid, qualify data. 3. If spiking solution is not valid, obtain fresh, certified spiking solution and reanalyze the sample and the associated matrix spikes. 4. If reanalysis of samples is not possible, qualify data. 5. Document actions taken.
SW8140	Organophosphorous pesticides	Equipment blank	Specified in RI work plans	Target compounds are not present at concentrations greater than the PDL.	If compounds found in equipment blank also are found in samples at similar concentrations, determine cause, evaluate effect on samples, and qualify data or resample if necessary.
		Field replicate	1 set per 10 samples	Within 30 percent RPD[1].	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control.
		Calibration curve	Established at 5 concentration levels.	Verification check standard is within 15 percent of the calibration curve regression.	1. Reanalyze check standards. 2. If similar results are obtained, recalibrate instrument. 3. Reanalyze samples processed since last time criteria were met. 4. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Method blank	1 per batch or 1 per 20 samples	Concentrations are less than the PDL of the analyte.	1. Determine source of contamination, that is, instrument, blank water or reagent.s 2. Take appropriate corrective action and document actions taken. 3. Reanalyze or reprepare batch. 4. If samples cannot be reanalyzed or reprepared, qualify data.
		DCS	1 duplicate per 20 samples	Recovery and precision are within limits established for matrix spike and matrix spike duplicate shown in Appendix B for 80 percent of the analytes.	1. Validate instrument parameters, sensitivity, and linearity. Correct problems and document actions taken. 2. Validate standards. 3. Validate DCS preparation. 4. Reprepare and reanalyze DCS and affected samples. 5. If repreparation of samples is not possible, qualify data. 6. Document actions taken.
		MS/MSD	1 set per 20 samples (Collector to provide duplicate samples)	See Appendix B for matrix spiking compounds, spike concentrations, and control limits.	1. Analyze spiking solution. 2. If spiking solution is valid, qualify data. 3. If spiking solution is not valid obtain fresh, certified spiking solution and reanalyze the sample and the associated matrix spikes. 4. If reanalysis of samples if not possible, qualify data. 5. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
SW8150	Chlorinated phenoxy acid herbicides	Equipment blank	Specified in RI work plans	Target compounds are not present at concentrations greater than the PDL.	If compounds found in equipment blank also are found in samples at similar concentrations, determine cause, evaluate effect on samples, and qualify data or resample if necessary.
		Field replicate	1 set per 10 samples	Within 30 percent RPD[1].	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control.
		Calibration curve	Established initially at 5 conc. levels, verified daily at mid level.	Verification check standard is within control limits specified in SW 846 3rd ed.	1. Reanalyze check standard. 2. If similar results are obtained, recalibrate instrument. 3. Reanalyze samples processed since last time criteria were met. 4. Document actions taken.
		Method blank	1 per batch or 1 per 20 samples, whichever is more frequent	Concentration is less than the PDL.	1. Determine source of contamination, that is, instrument, blank water, or reagents. 2. Take appropriate corrective action and document actions taken. 3. Reanalyze or reprepare batch. 4. If samples cannot be reanalyzed or reprepared, qualify data.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		DCS	1 duplicate per 20 samples	Recovery and precision are within limits established for matrix spike and matrix spike duplicate shown in Appendix B for 80 percent of the analytes.	1. Validate instrument parameters, sensitivity, and linearity. Correct problems and document actions taken. 2. Validate standards. 3. Validate DCS preparation. 4. Reprepare and reanalyze DCS and affected samples. 5. If repreparation of samples is not possible, qualify data. 6. Document actions taken.
		Surrogate spike	All blanks and samples	See Appendix B for surrogate spiking compounds, spike concentrations, and control limits.	1. Examine all QC (including but not limited to DCS, SCS, internal standards, and instrument check standards). 2. If surrogate in SCS is out of control, check quantification. If quantification is acceptable, reanalyze SCS. 3. If similar results are obtained from reanalysis, obtain fresh, certied SCS solution and reprepare QC samples and all affected samples. 4. If samples cannot be reprepared, qualify data. 5. If surrogate spike in SCS is acceptable but out of control for samples, validate preparation of samples. If no error or problems are discovered for sample preparation, qualify data. 6. If errors are discovered in preparation of samples, reprepare QC samples and all affected samples. 7. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		MS/MSD	1 set per 20 samples (Collector to provide duplicate samples).	See Appendix B for matrix spiking compounds, spike concentrations, and control limits.	1. Analyze spiking solution. 2. If spiking solution is valid, qualify data. 3. If spiking solution is not valid, obtain fresh, certified spiking solution and reanalyze the sample and the associated matrix spikes. 4. If reanalysis of samples is not possible, qualify data. 5. Document actions taken.
SW8240 E524.2	Volatile organic compounds	Trip blank	1 per shipment to laboratory	Target compounds are not present at concentrations greater than the PDL.	If compounds found in trip blank also are found in samples at similar concentrations, determine cause, evaluate effect on samples, and qualify data or resample if necessary.
		Ambient blank	1 per day	Target compounds are not present at concentrations greater than the PDL.	If compounds found in ambient blank also are found in samples at similar concentrations, determine cause, evaluate effect on samples, and qualify data or resample if necessary.
		Equipment blank	Specified in RI work plans	Target compounds are not present at concentrations greater than the PDL.	If compounds found in equipment blank also are found in samples at similar concentrations, determine cause, evaluate effect on sample, and qualify data or resample if necessary.
		Field replicate	1 set per 10 samples	Within 30 percent RPD[1].	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control.
		Tuning	Every 12 hours	Within limits of most recent CLP statement of work.	Adjust instrument parameters.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Calibration curve	Established initially at 5 conc. levels, verified daily at mid level.	Within limits of most recent CLP statement of work.	1. Recalibrate instrument. 2. Reanalyze samples processed since last time criteria were met. 3. Document actions taken.
		Calibration verification	At frequency specified in most recent CLP statement of work.	Within limits of most recent CLP statement of work.	1. Reanalyze check standard. 2. If similar results are obtained, recalibrate instrument. 3. Reanalyze samples since last time criteria were met. 4. Document actions taken.
		Internal standards	All blanks, standards, and samples.	The area of the internal standard in a sample must not vary by more than a factor of 2 (i.e. -50 to +100 percent) from the area of the same internal standard in the associated continuing calibration standard.	1. Check quantification. 2. Reanalyze extracts. 3. Reprepare and reanalyze sample(s). 4. If samples cannot be reprepared, qualify data. 5. Document actions taken.
		Method blank	1 per batch or 1 per 20 samples	Concentration if less than the PDL of the analyte except that common laboratory contaminates MeCl2, MEK, acetone, and toluene are less than five times the PDL.	1. Determine source of contamination, that is, instrument, blank water, or reagents. 2. Take appropriate corrective action and document actions taken. 3. Reanalyze or reprepare batch. 4. If samples cannot be reanalyzed or reprepared, qualify data.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		DCS	1 duplicate per 20 samples	Recovery and precision are within limits established for matrix spike and matrix spike duplicate shown in Appendix B for 80 percent of the analytes.	1. Validate instrument parameters, sensitivity, and linearity. Correct problems and document actions taken. 2. Validate standards. 3. Validate DCS preparation. 4. Reprepare and reanalyze DCS and affected samples. 5. If repreparation of samples is not possible, qualify data. 6. Document actions taken.
		Surrogate spike	All blanks and samples	See Appendix B for surrogate spiking compounds, spike concentrations, and control limits.	1. Examine all QC (including but not limited to DCS, SCS, internal standards, and instrument check standards). 2. If surrogate in SCS is out of control, check quantification. If quantification is acceptable, reanalyze SCS. If similar results are obtained from reanalysis, obtain fresh, certified SCS solution and reanalyze all QC and samples. 3. If surrogate in sample is out of control, examine area count for IS. iF IS area count is not within -50 to +100 percent of the IS area in the check standard, reanalyze samples.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		MS/MSD	1 set per 20 samples (Collector to provide duplicate samples)	See Appendix B for matrix spiking compounds, spike concentrations, and control limits.	1. Analyze spiking solution. 2. If spiking solution is valid, qualify data. 3. If spiking solution is not valid, obtain fresh, certified spiking solution and reanalyze the sample and the associated matrix spikes. 4. If reanalysis of samples if not possible, qualify data. 5. Document actions taken.
SW8270	Semivolatile organic compounds	Ambient blank	1 per day	Target compounds are not present at concentrations greater than the PDL.	If compounds found in ambient blank also are found in samples at similar concentrations, determine cause, evaluate effect on samples, and qualify data or resample if necessary.
		Equipment blank	Specified in RI work plans	Target compounds are not present at concentrations greater than the PDL.	If compounds found in equipment blank also are found in samples at similar concentrations, determine cause, evaluate effect on samples, and qualify data or resample if necessary.
		Field replicate	1 set per 10 samples	Within 30 percent RPD[1].	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control.
		Tuning	Every 12 hours	Within limits of EPA CLP latest statement of work.	Adjust instrument parameters.
		Calibration curve	Established initially at 5 conc. levels, verified daily at mid level.	Within limits of most recent CLP statement of work.	1. Reanalyze check standard. 2. If similar results are obtained, recalibrate instrument. 3. Reanalyze samples processed since last time criteria were met. 4. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Calibration verification	At frequency specified in most recent CLP statement of work.	Within limits of most recent CLP statement of work.	1. Reanalyze check standard. 2. If similar results are obtained, recalibrate instrument. 3. Reanalyze samples since last time criteria were met. 4. Document actions taken.
		Internal standards	All blanks, standards, and samples.	The area of the internal standard in a sample must not vary by more than a factor of 2 (i.e. -50 to +100 percent) from the area of the same internal standard in the associated continuing calibration standard.	1. Check quantification. 2. Reanalyze extracts. 3. Reprepare and reanalyze sample(s). 4. If samples cannot be reprepared, qualify data. 5. Document actions taken.
		Method blank	1 per batch or 1 per 20 samples	Concentrations are less than the PDL of the analyte except common laboratory contaminate bis-2-ethylhexylphthalate is less than five times the PDL.	1. Determine source of contamination, that is, instrument, blank water, or reagents. 2. Take appropriate corrective actions and document actions taken. 3. Reanalyze or reprepare batch. 4. If samples cannot be reanalyzed or reprepared, qualify data. 5. Document actions taken.
		DCS	1 duplicate per 20 samples	Recovery and precision are within limits established for matrix spike and matrix spike duplicate shown in Appendix B for 80 percent of the analytes.	1. Validate instrument parameters, sensitivity, and linearity. Correct problems and document actions taken. 2. Validate standards. 3. Validate DCS preparation. 4. Reprepare and reanalyze DCS and affected samples. 5. If reprepareation of samples is not possible, qualify data. 6. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Surrogate spike	All blanks, standards, and samples	See Appendix B for surrogate spiking compounds, spike concentrations, and control limits.	1. Examine all QC (including but not limited to DCS, SCS, internal standards, and instrument check standards). 2. If surrogate in SCS is out of control, check quantification. If quantification is acceptable, reanalyze SCS. 3. If similar results are obtained from reanalysis, and both the SCS and surrogate in the samples are similarly affected, obtain fresh, certified SCS solution and reprepare QC samples and all affected samples. 4. If only the SCS, and not the samples, is affected, report data and qualify. 5. If samples also are affected but cannot be reprepared, qualify data. 6. If surrogate spike in SCS is acceptable but out of control for samples, validate preparation of samples. If no error or problems are discovered for sample preparation, qualify data. 7. If errors are discovered in preparation of samples, reprepare QC samples and all affected samples. 8. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		MS/MSD	1 set per 20 samples (Collector to provide duplicate samples)	See Appendix B for matrix spiking compounds, spike concentrations, and control limits.	1. Analyze spiking solution. 2. If spiking solution is valid, qualify data. 3. If spiking solution is not valid, obtain fresh, certified spiking solution and reanalyze the sample and the associated matrix spikes. 4. If reanalysis of samples is not possible, qualify data. 5. Document actions taken.
	Explosives	Equipment blank (water only)	Specified in RI work plans	Concentration of analyte is less than the PDL.	If analyte found in equipment blank also is found in samples at similar concentration, determine cause, evaluate effect on samples, and qualify data or resample if necessary.
		Field replicate	1 set per 10 samples	Within 30 percent RPD for water samples[1] and within 40 percent RPD for soils.	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control.
		Calibration curve	Established initially at 5 points, verified every 10 samples	Verification check standard is within 10 percent of the calibration curve.	1. Recalibrate instrument. 2. If criteria are still exceeded, validate standards, reprepare, and, if necessary, recalibrate. 3. Reanalyze samples processed since last time criteria were met. 4. Document actions taken.
		Method blank	1 per batch or 1 per 20 samples	Concentration is less than the PDL of the analyte.	1. If analytes in blank exceed PDL, reprepare all blanks and samples. 2. Reanalyze batch. 3. If samples cannot be reprepared, continue with analysis of batch and qualify data. 4. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		DCS	1 duplicate per 20 samples	Values are within three SD of mean historical values or methodology control limits[2].	1. Analyze spiking solution. 2. If spiking solution results are acceptable, investigate possible error in preparation. 3. If no error is detected, qualify data. 4. If error is detected, reprepare and reanalyze samples and DCS. 5. Document actions taken.
		MS/MSD	1 set per 20 samples	Values are within three SD of mean historical values or methodology control limits.	1. Verify that the spike concentration is at least 50 percent of the unspiked concentration. 2. Verify that correct spiking solutions and amounts were used. 3. Check method blanks and DCS recovery. 4. Reanalyzed samples if laboratory error is suspected. 5. Document actions taken.
ASTM D2216	Soil moisture	Field replicate	1 set per 10 samples	Within 30 percent RPD.	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control.
		Analytical duplicate	1 per 20 samples	None established.	Not applicable.
E415.2	Total organic carbon	Field replicate	1 set per 10 samples	Within 30 percent RPD for water samples[1] and within 40 percent RPD for soils.	Use data to evaluate sample collection procedures. Resample if procedures are found to be out of control.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Calibration curve	Established daily at one point, verified with high and low standard.	Verification check standard is within 10 percent of the calibration curve.	1. Recalibrate instrument. 2. If criteria are still exceeded, validate standards, reprepare, and if necessary, recalibrate. 3. Reanalyze samples processed since last time criteria were met. 4. Document actions taken.
		DCS	1 duplicate per 20 samples	Recovery is within 91-109 percent and RPD is less than 20 percent.	1. Validate instrument parameters, sensitivity, and linearity. Correct problems and document actions taken. 2. Validate standards. 3. Validate DCS sample preparation. 4. Determine if samples require repreparation; reprepare if necessary. 5. If repreparation of samples is not possible, qualify data. 6. Document actions taken.
		MS/MSD	1 per 20 samples	Acceptance criteria are in Appendix B.	1. Verify that the spike concentration is at least 50 percent of the unspiked concentration. 2. Verify that correct spiking solutions and amounts were used. 3. Check method blanks and DCS recovery. 4. Reanalyze samples if laboratory error is suspected. 5. Document actions taken.
SW8280	Dioxins and furans	Tuning	Every 12 hours	Maximize high-mass sensitivity.	Adjust instrument parameters.

Summary of internal quality-control procedures--Continued

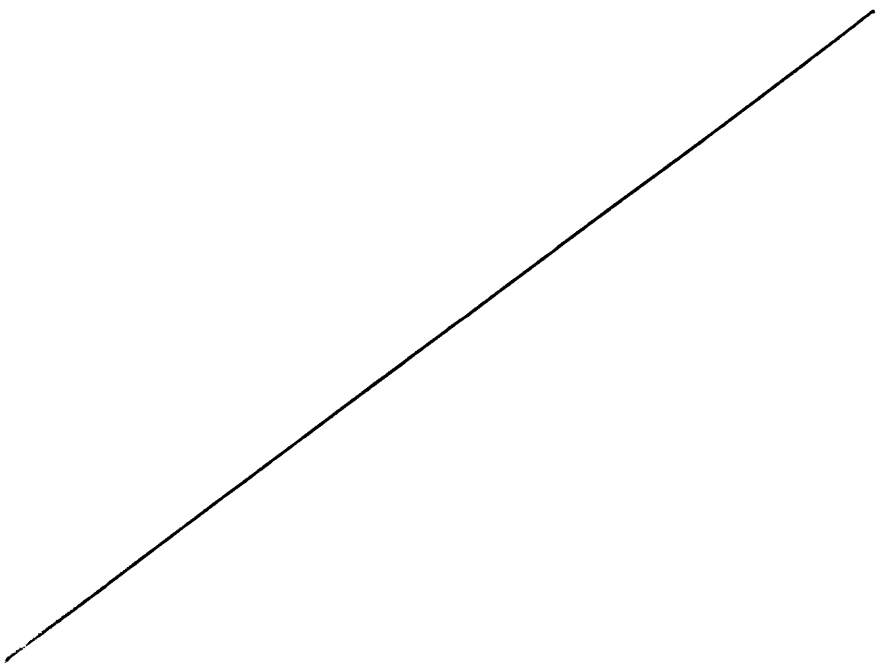
Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Calibration curve	Established initially at 15 points	RSD <15 percent	1. Validate instrument parameters. 2. Reanalyze standards. 3. Document actions taken.
		Routine calibration	Every 12 hours	1. Target analytes must be <30 percent difference from initial calibration. 2. Target secondary ion ratio limits in SW-846 must be met. 3. Minimum 25 percent resolution between 1,2,3,4 and 2,3,7,8 TCDD.	1. Validate instrument parameters. 2. Reanalyze check standards. 3. Recalibrate if needed. 4. Reanalyze all samples since las t acceptable standards. 5. Document actions taken.
		Method blank	1 per batch or 1 per 20 samples	Target compounds must be < target detection limits.	1. Determine source of contamination and correct. 2. Reanalyze or reprepare samples as appropriate. 3. If samples cannot be reprepared, qualify data. 4. Document actions taken.
		DCS	1 pair per batch or 1 per 20 samples	60 - 120 percent recovery	1. Validate instrument parameters. 2. Validate standards. 3. Validate DCS preparation. 4. Reanalyze DCS and affected samples. 5. If problem not corrected, qualify data. 6. Document actions taken.
		Surrogates	All blanks, standards, samples	None established	1. Examine all other QC, DCS, internal standards, check standards. 2. Verify sample preparation and analysis. 3. Document actions taken.

Summary of internal quality-control procedures--Continued

Analytical method	Property or analyte	Quality-control check	Frequency	Acceptance criteria	Corrective action
		Internal standards	All blanks, standards, samples	>40 percent recovery or >10:1 S/N ratio	1. Check quantification. 2. Reanalyze extracts. 3. Reprepare and reanalyze sample(s). 4. If samples cannot be reprepared, qualify data. 5. Document actions taken.
		Matrix spike and matrix spike duplicate	1 every 20 samples	60 - 120 percent recovery	1. Verify spiking solutions. 2. Check surrogates, internal standards, other QC to determine if repreparation is needed. 3. If samples cannot be reprepared, qualify data. 4. Document actions taken.
EPA (1990)	Acute toxicity	Replicates	4/treatment Ceriodaphnia, 2/treatment fathead minnows	None established	1. Retest if mortality exceeds 10 percent in control.

a. Field replicate water samples falling between 20 to 30 RPD will be flagged as falling within warning limits.

b. DCSs that fall between 2 and 3 standard deviations from historical values or methodology control limits will be considered within warning limits.



APPENDIX B

METHOD SUMMARIES AND ACCEPTANCE CRITERIA

I. E300.0 - Ion Chromatography (IC)

This is an ion chromatographic (IC) method applicable to determinations of anions in water samples (or water extracts). A small volume of sample (0.2 mL to 0.5 mL) is injected onto the anion separator column of an ion chromatograph. The anions of interest are separated and eluted with a sodium carbonate/sodium bicarbonate solution. The background conductivity of the eluant is reduced by a suppression device and the anions of interest are measured by a conductivity detector.

Sample Preparation

Water samples generally are analyzed directly on the instrument, with turbid samples being first filtered. Soil samples are extracted with deionized water (5:1 water:soil ratio) to produce a solution amenable to analysis. Thus, only the water-soluble fraction of the anions are determined.

Calibration

Standards are prepared from the sodium or potassium salts of each anion. The initial calibration curve is prepared using at least five different concentrations of each anion, in addition to a blank (deionized water). The multipoint calibration is re-established whenever the eluant or column is changed, after any major instrument or method changes, and whenever the calibration check standards fail to meet criteria.

A midrange check standard is analyzed daily before sample analysis, after every 10 samples, and at the end of the run. Deviations of more than ± 10 percent from the expected values require corrective action and possible recalibration.

A calibration blank is analyzed with every calibration check standard to monitor system cleanliness and carry-over. Values greater than the reporting limit require corrective action.

SW9040 - pH (water) and SW9045 - pH (soil)

An electrode sensitive to hydrogen ion concentration is immersed in the sample, producing a voltage between the sensing and reference elements of the electrode. This voltage is measured and converted to pH by a pH meter. Soils are prepared for pH analysis by mixing with deionized water in a 1:1 ratio.

Calibration

The meter is calibrated with commercially available buffers at pH 4 and pH 7. The calibration is checked by measuring a pH 10 buffer. After every 10 samples and at the end of the run, the calibration is checked against a solution of known pH (usually the DCS). All calibration checks must be within 2 percent of the expected value.

Internal QC

pH is a nonlinear function of the hydrogen ion concentration in the sample and is affected by the presence of many other components in the sample. Thus, it is not possible to perform matrix spikes for pH.

E120.1 - Specific Conductance

A cell consisting of two platinum sensing elements is immersed in the sample. The electrical resistance between the two elements is measured with a wheatstone bridge and converted to conductance. The reading is mathematically corrected to give the conductance at 25 °C. Soil samples are first extracted with deionized water (5:1 water:soil ratio) to produce a solution amenable to analysis.

Calibration

The instrument is calibrated with a potassium chloride solution that has a known conductance. Calibration is verified by measuring the conductance of the DCS, which is prepared independently. Verification is performed immediately after calibration, every 10 samples, and at the end of the run.

Internal QC

Specific conductance is a nonlinear function of the concentrations of the particular ions present in the sample. Thus, it is not possible to perform matrix spikes for pH.

E310.1 - Alkalinity

Alkalinity is comprised primarily of the hydroxide, carbonate, and bicarbonate ions present in a sample. Other bases (e.g. phosphate, borate, silicate) will contribute to the measured alkalinity if present in sufficient concentration. Alkalinity is measured by titrating a sample with dilute sulfuric acid to specified pH endpoints. The amount of acid consumed is directly proportional to the alkalinity of the sample. An empirical calculation allows the total alkalinity to be divided into hydroxide, carbonate, and bicarbonate forms. By convention, the results are reported as Calcium Carbonate. For soils, alkalinity is measured on a water extract of the sample (5:1 water:soil ratio), and only the soluble portion is determined.

The titrant is calibrated against a sodium hyroxide solution of known concentration. This in turn is calibrated against a solution of potassium hydrogen phthalate, which is a primary standard. All titrations are performed using a pH meter, which is calibrated as described under method SW9040.

E160.1 - Total Dissolved Solids (Waters)

Total dissolved solids are measured by filtering a known volume of sample through a specified filter, evaporating the water, and drying the residue that remains at 180 °C. The residue is then weighed and reported as total dissolved solids (TDS).

All balances are serviced and calibrated at least annually to maintain calibration. Class "S" weights are used to verify the calibration of the balance each day it is used. Oven temperatures are monitored with thermometers that are calibrated annually against an NIST certified thermometer.

D2216 - Moisture Content (Soils)

A portion of the sample is weighed and then dried in an oven at 105 °C. The dried sample is weighed again and the moisture content determined by difference.

All balances are serviced and calibrated at least annually to maintain calibration. Class "S" weights are used to verify the calibration of the balance each day it is used. Oven temperatures are monitored with thermometers that are calibrated annually against an NIST certified thermometer.

II. SW 6010 - Total Metals (ICP)

Inductively Coupled Argon Plasma/Optical Emission Spectroscopy (ICP/OES or ICP) simultaneously determines the concentration of a number of elements in a sample using atomic emission spectroscopy. Prior to analysis, the appropriate digestion techniques are carried out. An aerosol of the prepared sample is introduced into a radio-frequency inductively argon plasma, which produces element-specific atomic emission spectra. The particular wavelengths produced are characteristic of the elements present in the sample, and the intensity of the light is proportional to concentration. The light produced by the plasma is dispersed through a diffraction grating and the intensities at specific wavelengths are measured with photomultiplier tubes. The intensities are converted to concentration by means of the calibration curve and are corrected for background and interelement interferences.

Sample Preparation

Methods SW3010 (water samples) and SW3050 (soil samples) are used to prepare samples for total metals analysis by ICP. These methods use an acid digestion to solubilize and stabilize the metals in solutions that are amenable to analysis on the instrument.

Calibration

The instrument is calibrated daily at two points, one of which is a multi-element calibration standard and the other a calibration blank (the multi-element standard may be in several solutions to prevent precipitation of metals). After the initial standardization has been performed, the mixed standard is immediately re-analyzed. If the results are not within 5 percent of the expected value, corrective action must be performed. An initial calibration verification (ICV) and an initial cali-

bration blank (ICB) are then analyzed. The ICV standard is from a source different than that used to establish initial calibration. The ICV must be within 10 percent of expected response and the ICB must be less than the reporting limit for each element.

After every 10 samples and at the end of the run, a continuing calibration verification (CCV) and continuing calibration blank (CCB) are analyzed. The CCV must be within 10 percent of the expected response and the CCB must be less than the reporting limit. If these criteria are not met for any element, all samples dating back to the last acceptable CCV/CCB must be reanalyzed for that element.

Interelement Correction Factors (IECs) are established annually for each instrument. Interelement check standards are then analyzed at the beginning and end of each ICP run to verify that interelement and background correction factors have remained constant. The IEC check consists of two solutions. The first (ICSA) contains only the interfering elements (Al, Fe, Ca, Mg) and is used to ensure that the IECs will not produce false positive results. The second (ICSAB) contains target analytes in addition to the interfering elements and ensures the accuracy of the IECs. Results outside the criteria in table 2 require corrective action and reanalysis of any affected samples.

Instrument detection limits (IDLs) are determined quarterly to ensure instrument sensitivity. The IDL is statistically calculated from replicate analyses of a low-concentration standard. The standard used for the IDL does not go through the sample preparation procedure.

SW 7060, SW 7421, SW 7740, SW 7841 - Total Metals (GFAA)

Graphite Furnace Atomic Absorption Spectroscopy (GFAA) is used to measure low concentrations of individual elements in a sample. After sample preparation, matrix modifiers are added to control interferences. A small portion of the sample is then injected into the graphite furnace. The furnace cycles through a temperature program that dries the sample, drives off interferences, and finally atomizes the sample at high temperature. A specially designed lamp emits the spectrum of the element of interest, which passes through the graphite furnace and is absorbed by the atoms in the furnace. The intensity of the light is measured with a photomultiplier tube, and the amount absorbed is proportional to the concentration of the element in the sample.

Sample Preparation

Methods SW3020 (water samples) and SW3050 (soil samples) are used to prepare samples for total metals analysis by methods SW7421 (lead) and SW7841 (thallium). These methods use an acid digestion to solubilize and stabilize the metals in solutions that are amenable to analysis on the instrument. Methods SW7060 (arsenic) and SW7740 (selenium) contain specialized variations of the digestion method that have been optimized for these particular elements.

Calibration

Daily calibration consists of a minimum of three standards plus a blank. A linear calibration curve is then calculated. The correlation coefficient must be at least 0.995. An Initial Calibration Verification (ICV) and Initial Calibration Blank (ICB) are analyzed immediately after the daily calibration. The ICV is an independent standard that serves to verify the accuracy of the calibration curve. A Continuing Calibration Verification standard (CCV) and Continuing Calibration Blank (CCB) are analyzed after every 10 samples and at the end of the run. All calibration verifications must be within 10 percent of the expected value, and calibration blanks must be less than the reporting limit.

Internal QC

Every sample analyzed by GFAA receives an analytical spike. This is a spike performed at the instrument to detect the presence of matrix interferences. The spike recovery must be 80 to 120 percent. Corrective action depends on spike recovery and whether the analyte was detected in the sample (refer to table 3).

SW 7470 (Water) and SW 7471 (Soil) - Total Mercury (CVAA)

Cold Vapor Atomic Absorption Spectroscopy (CVAA) takes advantage of the volatility of elemental mercury. The sample is digested with a combination of reagents, which convert the various forms of mercury into mercury ions in a solution that is amenable to analysis. The mercury ions are then reduced to elemental mercury, which is sufficiently volatile to be sparged from the sample into a cell. A specially designed lamp emits the spectrum of mercury, which passes through the cell and is absorbed by the mercury vapor. The amount of light absorbed is measured with a photomultiplier tube and is proportional to the concentration of mercury in the sample.

Calibration

Daily calibration consists of a minimum of four standards and a blank. A linear calibration curve is then calculated. The curve must have a slope between 70 and 113 mV per ppm mercury and a correlation coefficient of at least 0.995. An Initial Calibration Verification (ICV) and Initial Calibration Blank (ICB) are analyzed immediately after the daily calibration. The ICV is an independent standard that serves to verify the accuracy of the calibration curve. A Continuing Calibration Verification standard (CCV) and Continuing Calibration Blank (CCB) are analyzed after every 10 samples and at the end of the run. All calibration verifications must be within 10 percent of the expected value, and calibration blanks must be less than the reporting limit.

III. SW 8080 - Organochlorine Pesticides and PCBs

Method SW8080 tests soil and liquid samples for organochlorine pesticides and polychlorinated biphenyl (PCBs) mixtures. Samples are prepared as described below and analyzed on a gas chromatograph equipped with an electron capture detector.

Sample Preparation

Water samples are extracted at a neutral pH with methylene chloride by methods SW3510 or SW3520. Method SW3510 is a separatory funnel extraction technique and SW3520 is a continuous liquid-liquid extraction. Soil samples are extracted with methylene chloride and acetone using method SW3550, a sonication extraction procedure. Extracts are solvent exchanged into hexane and receive cleanup procedures to remove interferences as deemed necessary for the sample.

Calibration

Calibration involves initial five point initial calibrations for most target analytes. For the aroclors, toxaphene, technical chlordane, and certain of the Appendix IX pesticides, a single point calibration at or slightly above the method reporting limit is established initially. If any of these analytes are detected in a sample, a five point calibration curve is established and the sample is reanalyzed and quantitated against the five-point curve.

The initial calibration is performed upon instrument setup, any major system change, and when daily or continuing standards no longer meet criteria. For each target analyte at each concentration level, calibration factors are calculated. The average calibration factor is calculated as:

$$CF_{avg} = \frac{CF_1 + CF_2 + CF_3 + \ldots CF_n}{n}$$

where,

CF_{avg} = average calibration factor,

CF_n = calibration factor for n calibration standard of the initial calibration, and

n = number of standards used for initial calibration.

For a valid calibration curve, the % Relative Standard Deviation (%RSD) for the calibration factors must be less than 20 percent for each compound. %RSD is calculated as follows:

$$\%RSD = \frac{SD}{CF_{avg}} \times 100\%$$

where,

%RSD = percent relative standard deviation,

CF_{avg} = average response factor, and

SD = standard deviation of the CFs for a compound.

The Pesticide Evaluation Mixture is analyzed at the beginning of each run to check for degradation of endrin and 4,4'-DDT caused by a dirty injection port or column. Degradation of either

compound by more than 20 percent requires that system maintenance be performed before proceeding with the analysis.

A Continuing Calibration Standard (the midpoint standard from the five-point curve) is analyzed daily before any samples, after every 10 samples, and at the end of the run to ensure that instrument sensitivity does not change. The calibration factor for each analyte must be within 15 percent of the initial calibration curve.

Sample Identification and Quantitation

For single chromatographic peak analytes, any positive hit on the primary analytical column must be confirmed by analysis on a second dissimilar column. The presence of the target analyte is confirmed by agreement of the results for the two dissimilar columns. Quantitation is performed relative to the initial calibration.

Several target analytes of the method are not pure compounds but mixtures of chemicals (the seven aroclor (PCB) mixtures, toxaphene, and technical chlordane). These target analytes produce chromatograms with multiple peaks in recognizable patterns and identification is based primarily on the comparison of a sample pattern to that of a standard.

IV. SW 8150 - Chlorinated Herbicides

Method 8150 provides extraction, esterification, and gas chromatographic conditions for the analysis of chlorinated acid herbicides. The esters are hydrolyzed to the corresponding acid forms with potassium hydroxide and extraneous organic material is removed by a solvent wash. After acidification, the acids are extracted with solvent and converted to their methyl esters using diazomethane. After excess reagent is removed, the esters are determined by gas chromatography employing an electron capture detector.

Calibration

A multipoint initial calibration is performed to establish the linear range of the analytical system. The initial calibration is performed upon instrument setup, any major system change, and when daily or continuing standards no longer meet criteria. A minimum of five concentration levels containing the analytes of interest are analyzed. A response factor (RF) is tabulated for each compound:

$$RF = \frac{A_x}{C_x}$$

where,

RF = response factor,

A_x = area for the compound being measured, and

C_x = concentration of the compound being measured.

The average response factor (RFavg) is calculated for each compound using the RF from each calibration point:

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 \ldots + RF_n}{n}$$

where,

RF_{avg} = average response factor,

RF_n = response factor for n calibration standard of the initial calibration, and

n = number of standards used for initial calibration.

The Percent Relative Standard Deviation (%RSD) must be less than 15 percent and is calculated as follows:

$$\%RSD = \frac{SD}{RF_{avg}} \times 100\%$$

where,

%RSD = percent relative standard deviation,

RF_{avg} = average response factor, and

SD = standard deviation of the RFs for a compound.

A continuing calibration standard (CCS), usually at a midlevel concentration, is analyzed at the beginning and end of each run and after every 10 samples. The CCS RF must not differ from the RFavg of the initial calibration by more than 20 percent. Percent difference (%D) is calculated as follows:

$$\%D = \frac{RF_{avg} - RF_c}{RF_{avg}} \times 100\%$$

where,

%D = percent difference,

RF_{avg} = average response factor from initial calibration, and

RF_c = response factor from CCS.

When all CCS criteria are met, sample analyses may proceed.

Sample Identification and Quantitation

Any positive hit on the primary analytical column must be confirmed by analysis on a second dissimilar column. The presence of the target analyte is confirmed by agreement of the results for the two dissimilar columns. Quantitation is performed relative to the initial calibration.

Internal QC

A methylation standard is analyzed with every batch of samples to ensure that esterification is complete. Recoveries of less than 80 percent may indicate a problem with the sample preparation technique or reagents used.

V. SW 8240 - Volatile Organics by GC/MS

This method is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure and is used to determine volatile organic compounds in a variety of matrices, including water and soil. The volatile compounds are extracted and introduced into the gas chromatograph by the purge-and-trap method. The components are separated via the gas chromatograph and detected using a mass spectrometer which provides both qualitative and quantitative information.

Preparation

Volatile compounds in water or low-level contaminated soils can be introduced directly into the gas chromatograph by the purge-and-trap method (SW 5030). Medium-level contaminated soils may require methanolic extraction, as described in method SW 5030, prior to purge-and-trap.

Calibration

The mass spectrometer's tuning is checked prior to any sample or calibration standard analysis and every 12 hours. A standard containing 50 nanograms (ng) of 4-Bromofluorobenzene (BFB) is injected into the GC/MS. The resulting mass spectrum must meet all the criteria listed in table 2 before standard analysis may proceed.

A multipoint initial calibration is performed to establish the linear range of the analytical system. The initial calibration is performed upon instrument setup, any major system change, and when daily or continuing standards no longer meet criteria. A minimum of five concentration levels containing the analytes of interest and internal standards are analyzed. A relative response factor (RF) is tabulated for each compound relative to the internal standard whose retention time is closest to the compound being measured:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where,

RF = response factor,

A_x = area of characteristic ion for the compound being measured,

C_{is} = concentration of the specific internal standard, and

A_{is} = area of characteristic ion for the specific internal standard,

C_x = concentration of the compound being measured.

The average relative response factor (RF_{avg}) is calculated for each compound using the RF from each calibration point:

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 \ldots + RF_n}{n}$$

where,

RF_{avg} = average response factor,

RF_n = response factor for n calibration standard of the initial calibration, and

n = number of standards used for initial calibration.

Before the initial calibration can be accepted, system performance check compounds (SPCC) and calibration check compounds (CCC) are evaluated. The SPCC are checked for a minimum average relative response factor. The five volatile SPCC compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chorobenzene. The minimum acceptable average relative response factor is 0.300 (0.250 for bromoform). The CCC are checked for the percent relative standard deviation (%RSD) of RFs in the initial calibration. The volatile CCC are 1,1-dichloroethane, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride. The %RSD of the RF for each of these compounds must be $\leq$30 percent.

%RSD is calculated as follows:

$$\%RSD = \frac{SD}{RF_{avg}} \times 100\%$$

where,

%RSD = percent relative standard deviation,

RF_{avg} = average response factor, and

SD = standard deviation of the RFs for a compound.

A continuing calibration standard (CCS), usually at a midlevel concentration, is analyzed after instrument tuning and every 12 hours. The CCS is evaluated by criteria applied to the SPCC and CCC given above. The SPCC RF must be $\geq$0.300 (0.250 for bromoform); the CCC RF must not differ from the RF_{avg} of the initial calibration by more than 25 percent. Percent difference (%D) is calculated as follows:

$$\%D = \frac{RF_{avg} - RF_c}{RF_{avg}} \times 100\%$$

where,

%D = percent difference,

RF_{avg} = average response factor from initial calibration, and

RF_c = response factor from CCS.

The internal standard responses and retention times in the continuing calibration standard must be evaluated. If any internal standard retention time changes by more than 30 seconds from the last calibration check, the system must be checked for malfunctions and corrections made as necessary. If the extracted ion current profile (EICP) area for any of the internal standards changes by a factor of more than two from the last daily calibration standard check, the system must be checked for malfunctions and corrections made as necessary. When all CCS criteria are met, sample analyses may proceed. Sample quantitation is performed based on the CCS RF.

VI. SW 8270 - Semivolatile Organic Compounds by GC/MS

This method can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols.

Certain Appendix IX compounds are either not reliably recoverable by this method, or standards are not available (Aramite, Dibenz(a,j)acridine, Dimethoate, Famphur, Hexachlorophene, 4-Nitroquinoline-n-oxide, p-Phenylenediamine). In order to detect their presence, a library search is made for these compounds.

Diphenylamine cannot be distinguished from N-Nitroso-diphenylamine because of decomposition in the injection port. 3-Methylphenol and 4-Methylphenol cannot be distinguished because of their identical retention times and mass spectra.

Sample Preparation

Prior to using this method, samples must be prepared using the appropriate sample preparation method: for soil samples, sonication extraction (SW3550) is used and for water samples, separatory funnel (SW 3510) or continuous liquid/liquid extraction (SW3520) are used.

Calibration

The mass spectrometer's tuning is checked prior to any sample or calibration standard analysis and every 12 hours. A standard containing 50 ng of Decafluorotriphenylphosphine (DFTPP) is injected into the GC/MS. The resulting mass spectrum must meet all the criteria listed in Table 2 before standard analysis may proceed.

Method SW846 requires that initial and continuing calibrations be analyzed, evaluated, and accepted before sample analysis may proceed. A multipoint initial calibration is performed to establish the linear range of the analytical system. The initial calibration is performed upon instrument setup, any major system change, and when daily or continuing standards no longer meet criteria. A minimum of five concentration levels containing the analytes of interest and internal

standards are analyzed. A relative response factor (RF) is tabulated for each compound relative to the internal standard whose retention time is closest to the compound being measured:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where,

RF = relative response factor,

A_x = area of characteristic ion for the compound being measure,

C_{is} = concentration of the specific internal standard, and

A_{is} = area of characteristic ion for the specific internal standard,

C_x = concentration of the compound being measured.

The average relative response factor (RF_{avg}) is calculated for each compound using the RF from each calibration point:

$$RF_{avg} = \frac{RF_1 + RF_2 + RF_3 \ldots + RF_n}{n}$$

where,

RF_{avg} = average relative response factor,

RF_n = response factor for n calibration standard of the initial calibration, and

n = number of standards used for initial calibration.

Before the initial calibration can be accepted, system performance check compounds (SPCC) and calibration check compounds (CCC) are evaluated. The SPCC are checked for a minimum average relative response factor. The four semivolatile SPCC compounds are N-nitrous-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. The minimum acceptable average relative response factor is 0.050. The CCC are checked for the percent relative standard deviation (%RSD) of RFs in the initial calibration. The semivolatile CCC are acenaphthene, 1,4-dichlorobenzene, hexachlorobutadiene, N-nitroso-di-phenylamine, di-n-octylphthalate, fluoranthene, benzo(a)pyrene, 4-chloro-3-methylphenol, 2,4-dichlorophenol, 2-nitrophenol, phenol, pentachlorophenol, and 2,4,6-trichlorophenol. The %RSD of the RF for each of these compounds must be $\leq$ 30 percent.

%RSD is calculated as follows:

$$\%\text{RSD} = \frac{SD}{RF_{avg}} \times 100\%$$

where,

%RSD = percent relative standard deviation,

RF_{avg} = average response factor, and

SD = standard deviation of the RFs for a compound.

A continuing calibration standard (CCS), usually at a midlevel concentration, is analyzed every 12 hours and after instrument tuning. The CCS is evaluated by criteria applied to the SPCC and CCC given above. The SPCC RF must be ≥ 0.050; the CCC RF must not differ from the RF_{avg} of the initial calibration by more than 30 percent. Percent difference (%D) is calculated as follows:

$$\%D = \frac{RF_{avg} - RF_c}{RF_{avg}} \times 100\%$$

where,

%D = percent difference,

RF_{avg} = average relative response factor from initial calibration, and

RF_c = relative response factor from CCS.

The internal standard responses and retention times in the continuing calibration standard must be evaluated. If any internal standard retention time changes by more than 30 seconds from the last calibration check, the system must be checked for malfunctions and corrections made as necessary. If the extracted ion current profile (EICP) area for any of the internal standards changes by a factor of more than two from the last daily calibration standard check, the system must be checked for malfunctions and corrections made as necessary. When all CCS criteria are met, sample analyses may proceed. Sample quantitation is performed based on the CCS RF.

VIII. SW 8330 - Explosives

Method 8330 provides high performance liquid chromatographic (HPLC) conditions for the detection of parts per billion (ppb)levels of certain explosives and related compounds. Water samples are first treated with sodium chloride to reduce the solubility of the target analytes and extracted with acetonitrile. Soil samples are extracted with acetonitrile using sonication. Aliquots of the extract are injected into a HPLC, and compounds in the effluent are detected by an ultraviolet (UV) detector.

Calibration

A five point calibration curve is established for each analyte upon initial instrument setup, after any major system change, and when the daily standard checks fail to meet criteria. The average calibration factor for each compound is calculated as:

$$CF_{avg} = \frac{CF_1 + CF_2 + CF_3 \ldots CF_n}{n}$$

where,

CF_{avg} = average calibration factor,

CF_n = calibration factor for n calibration standard of the initial calibration, and

n = number of standards used for initial calibration.

For a valid calibration curve, the% Relative Standard Deviation (%RSD) for the calibration factors must be less than 25 percent for each compound.%RSD is calculated as follows:

$$\%RSD = \frac{SD}{CF_{avg}} \times 100\%$$

where,

%RSD = percent relative standard deviation,

CF_{avg} = average response factor, and

SD = standard deviation of the CFs for a compound.

A midpoint standard is analyzed on a daily basis prior to the analysis of any samples, after every 10 samples, and at the end of the run. The response must be within 25 percent of that expected from the initial calibration.

IX. Polychlorinated Dibenzodioxins and Polychlorinated Dibenzofurans by GC/MS

This method is applicable to the determination of tetra-, penta-, hexa-, hepta-, and octa-chlorinated dibenzo-p-dioxins (PCDDs) and dibenzo-furans (PCDFs). This procedure uses a matrix-specific extraction, analyte-specific cleanup, and high resolution capillary column gas chromatography/low resolution mass spectrometry (HRGC/LRMS). Method SW8280 requires that six isotopically labelled (carbon 13) analogs of target analytes (internal standards) be spiked into each sample before extraction. These analogs elute and behave as target analytes without interfering with the analysis. Target analytes are quantitated relative to the internal standard and their calculated concentrations are thereby compensated for extraction efficiency.

The sensitivity of the method is dependent upon the level of interference within a given matrix. Target quantification levels for target analytes are 0.4 ppb in solid samples and 4 parts per trillion (ppt) in water. Actual values have been shown to vary by homologous series and, to a lesser degree, by individual isomer. The detection limit for each of the chlorination levels and each congener is calculated from the noise level present in the elution window and the height of the chromatographic peak of the internal standard. Both values are measured by the GC/MS data system and the result of the calculation is a detection limit that is specific to the homologous series and sample. There is a three-tiered approach to reporting and detection limits:

1. In the absence of target analytes, a sample-specific estimated detection limit (EDL) is calculated based on the signal-to-noise ratio. The target analyte is then reported as "not detected" (ND) at the EDL.

2. When target analytes are found, the lower calibration limit is used as the detection limit. If the concentration found is above this value, it is reported without additional qualifiers. If the concentration found is less than the lower calibration limit, it is qualified as such.

3. If a peak is present that meets the signal-to-noise criteria but not all of the other identification criteria (e.g. retention time, ion ratio, absence of diphenyl ethers, and analyst judgment), the Estimated Maximum Possible Concentration (EMPC) is calculated based on the ion peak. The target analyte is reported as "not detected" (ND) at a detection limit equal to the EMPC and the result is qualified as an EMPC.

The assessment of matrix effects on method performance is assessed using the isotopically labeled analogs. These are spiked into every sample and matrix effects can therefore be evaluated from the recovery of the analogs for each sample. Acceptance of the sample analysis is generally controlled on this basis rather than a matrix spike/matrix spike duplicate.

Dioxins and Furans

Component	Method	Reporting Limit Water (nanograms per liter)	Reporting Limit Soil (nanograms per gram)
TCDFs (total)	SW8280	4	0.4
2,3,7,8-TCDF	SW8280	4	0.4
PeCDFs (total)	SW8280	20	2
1,2,3,7,8-PeCDF	SW8280	20	2
2,3,4,7,8-PeCDF	SW8280	20	2
HxCDFs (total)	SW8280	20	2
1,2,3,4,7,8-HxCDF	SW8280	20	2
1,2,3,6,7,8-HxCDF	SW8280	20	2
2,3,4,6,7,8-HxCDF	SW8280	20	2
1,2,3,7,8,9-HxCDF	Sw8280	20	2
HpCDFs (total)	SW8280	20	2
1,2,3,4,6,7,8-HpCDF	Sw8280	20	2
1,2,3,4,7,8,9-HpCDF	SW8280	20	2
OCDF	SW8280	40	4
TCDDs (total	SW8280	4	0.4
2,3,7,8-TCDD	SW8280	4	0.4
PeCDDs (total)	SW8280	20	2
1,2,3,7,8-PeCDD	SW8280	20	2
HxCDDs (total)	SW8280	20	2
1,2,3,4,7,8-HxCDD	SW8280	20	2
1,2,3,6,7,8-HxCDD	SW8280	20	2
1,2,3,7,8,9-HxCDD	SW8280	20	2
HpCDDs (total)	SW8280	20	2
1,2,3,4,6,7,8-HpCDD	SW8280	20	2
OCDD	SW8280	40	4

SW8280 specifies that sample-specific estimated detection limits (EDLs) be calculated and reported. Target reporting limits are given above.

TABLE 2. SUMMARY OF CALIBRATION PROCEDURES

Parameter	Method	Calibration	Frequency	Acceptance Criteria	Corrective Action
Common Anions	E300.0	Initial Calibration (minimum 5 points plus blank)	Initially and as required	RSD ≤ 20%	1) Evaluate system 2) Recalibrate
		Initial/Continuing Calibration Standard	Daily before sample analysis, every 10 samples, end of run	90-100% Recovery	1) Evaluate system 2) Reanalyze standard 3) Recalibrate if required
		Initial/Continuing Calibration Blank	Daily before sample analysis,	< Reporting Limit	1) Reanalyze blank 2) Clean system if required 3) Assess impact on data 4) Reanalyze affected samples
pH	SW9040 (w),	Initial Calibration	Daily before analysis	See Calibration Verification	N/A
	SW9045 (s)	(pH 4 and 7)			
		Calibration Verification (pH 10)	After initial calibration	98-102% Recovery	1) Perform maintenance 2) Recalibrate
		Continuing Calibration	Every 10 samples, end of run	98-102% Recovery	1) Evaluate system 2) Reanalyze standard 3) Recalibrate if required 4) Reanalyze affected samples
Specific Conductance	E120.1	Initial Calibration	Daily before analysis	DCS Within Limits	1) Perform maintenance 2) Recalibrate
		Continuing Calibration	Every 10 samples, end of run	95-105%	1) Evaluate system 2) Reanalyze standard 3) Recalibrate if required 4) Reanalyze affected samples

TABLE 2. SUMMARY OF CALIBRATION PROCEDURES --Continued

Parameter	Method	Calibration	Frequency	Acceptance Criteria	Corrective Action
Moisture Content	D2216	Balance Calibration	Daily before sample analysis	99.5-100.5%	Perform maintenance
Total Dissolved Solids (TDS)	E160.1	Balance Calibration	Daily before analysis	99.5-100.5%	Perform maintenance
Alkalinity	E310.1	Initial Calibration	Daily before analysis (pH 4 and 7)	See Calibration Verification	N/A
		Calibration Verification (pH 10)	After initial calibration	98-102% Recovery (pH)	1) Perform maintenance 2) Recalibrate
		Continuing Calibration	Every 10 samples, end of run	98-102% Recovery (pH)	1) Evaluate system 2) Reanalyze standard 3) Recalibrate if required 4) Reanalyze affected samples
Metals, Total	SW7060, SW7421, SW7740,	Calibration Blank + Three point calibration	Daily at beginning of run	Correlation Coefficient $\geq$ 0.995	1) Evaluate system 2) Recalibrate
	SW7841	ICV (Initial Calibration Verification	Immediately after standardization	90-110% Recovery	1) Reanalyze ICV 2) Recalibrate if required
		CCV (Continuing Calibration Verification	Every 10 samples and end of run	90-110% Recovery	1) Reanalyze CCV 2) Recalibrate if required 3) Reanalyze affected samples
		ICB/CCB (Initial/Continuing Calibration Blank)	After each ICV and CCV	< Reporting Limit	1) Reanalyze ICB/CCB 2) Clean system if required 3) Reanalyze affected samples
	SW7470, SW7471	Calibration Blank + Four point calibration	Daily at beginning of run	1) Correlation Coefficient $\geq$ 0.995 2) Slope = 70-113 mV/ppm	1) Check instrument settings 2) Recalibrate

TABLE 2. SUMMARY OF CALIBRATION PROCEDURES --Continued

Parameter	Method	Calibration	Frequency	Acceptance Criteria	Corrective Action
		ICV	Immediately after standardization	90-110% Recovery	1) Reanalyze ICV 2) Recalibrate if required
		CCV	Every 10 samples and end of run	90-110% Recovery	1) Reanalyze CCV 2) Recalibrate if required 3) Reanalyze affected samples
		ICB/CCB	After each ICV and CCV	≤ Reporting limit	1) Reanalyze ICB/CCB 2) Clean system if required 3) Reanalyze affected samples
	SW6010	Linearity Check	Quarterly	Linear range = highest standard with reading 95-105% of true value	Dilute samples with analytes higher than Linear Range
		IECs (Interelement Corrections)	Annually	Affected elements <IDL	Recalculate IECs
		IDL (Instrument Detection Limit	Quarterly	< Reporting Limit	1) Check calculations 2) Perform maintenance 3) Re-run IDL
		Mixed calibration standards	Daily prior to analysis	95-105% Recovery	Recalibrate
		ICV	Immediately after calibration	90-110% Recovery	1) Reanalyze ICV 2) Recalibrate if required
		ICB	After each ICV	< Reporting Limit	1) Reanalyze ICB/CCB 2) Clean system if required 3) Recalibrate if required
		ICSA (Interference Check Standard A)	After ICV, every 8 hours, and after last sample	1) 80-120% (Al, Fe, Ca, Mg) 2) ≤ Reporting Limit for other elements	1) Reanalyze 2) Verify calibration 3) Adjust IECs and recalibrate if required
		ICSAB (Interference Check Standard A/B)	After each ICSA	80-120% Recovery	Same as ICSA

TABLE 2. SUMMARY OF CALIBRATION PROCEDURES --Continued

Parameter	Method	Calibration	Frequency	Acceptance Criteria	Corrective Action
		CCV	10%, plus end of run	90-110% Recovery	1) Reanalyze CCV 2) Recalibrate if required 3) Reanalyze affected samples
		CCB	After each CCV	<Reporting Limit	1) Reanalyze ICB/CCB 2) Clean system if required 3) Reanalyze affected samples
Pesticides and PCBs	SW8080	5 point calibration	Initially and as required	RSD < 20%	1) Evaluate system 2) Recalibrate
		Single pt. calibration: PCBs, diallate, isodrin, kepone, chlordane, chlorbenzilate	Initially and as required	All samples < Reporting Limit for these analytes	1) Run 5 point calibration for affected analyte(s) 2) Requantitate samples
		PEM (Pesticide Evaluation Mixture)	Beginning of each sequence	> 80% Recovery	Perform system maintenance
		Calibration Check Standard	Daily before sample analysis; analysis; every 10 samples; end of run	85-115% Recovery	1) Evaluate system 2) Reanalyze standard 3) Assess impact on data 4) Recalibrate if necessary 5) Reanalyze affected samples
Herbicides	SW8150	6 point calibration	Initially and as required	RSE < 15%	1) Evaluate system 2) Recalibrate
		Calibration Check Standard	Daily before sample analysis, every 10 samples, end of run	80-120% Recovery	1) Evaluate system 2) Reanalyze standard 3) Recalibrate if necessary 4) Reanalyze affected samples

TABLE 2. SUMMARY OF CALIBRATION PROCEDURES --Continued

Parameter	Method	Calibration	Frequency	Acceptance Criteria	Corrective Action
Volatile Organics	SW8240	5 point calibration	Initially and as required	Minimum Response Factors for SPCC compounds: Chloromethane ≥ 0.30 1,1-Dichloroethenane ≥ 0.30 Bromoform ≥ 0.25 Chlorobenzene ≥ 0.30 1,1,2,2-Tetrachloroethane ≥ 0.30 RSD ≤ 30% for CCC Compounds (Vinyl Chloride, Ethyl Benzene 1,1-Dichloroethane, Chloroform, !,2-Dichloropropane, Toluene)	1) Evaluate system 2) Recalibrate
		BFB tuning standard	Start of run, every 12 hours	Mass/Criterion: 50: 15-40% of mass 95 75: 30-60% of mass 95 95: Base peak 96: 5-9% of mass 95 173: <2% of mass 174 174: >50% of mass 95 175: 5-9% of mass 174 176: 95-101% of mass 174 177: 5-9% of mass 176	1) Retune instrument 2) Reanalyze BFB
		Continuing Calibration Standard	Daily before sample analysis;analysis every 12 hours	Meet Min. Response Factors for SPCCs	1) Evaluate system 2) Reanalyze standard 3) Recalibrate if necessary 4) Reanalyze affected samples

TABLE 2. SUMMARY OF CALIBRATION PROCEDURES --Continued

Parameter	Method	Calibration	Frequency	Acceptance Criteria	Corrective Action
		Internal Standard	Every sample	Area = 50-200% of daily Cont. Cal. Standard	1) Check integration 2) Reanalyze standard 3) Assess impact on data 4) Reanalyze sample if required
Semivolatile Organics	SW8270	5 point calibration	Initially and as required	Minimum Response Factor of 0.05 for SPCC compounds: N-Nitroso-di-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol, 4-Nitrophenol	1) Evaluate system 2) Recalibrate
				RSD $\leq$ 30% for CCC Compounds Acenaphthene, 1,4-Dichlorobenzene, Hexachlorobutadiene, N-Nitroso-diphenylamine, Di-n-octyl phthalate, Fluoranthene, Benzo(a)pyrene, 4-Chloro-3-methylphenol, 2,4-Dichlorophenol, 2-Nitrophenol, Phenol, Pentachlorophenol, 2,4,6-Trichlorophenol	

TABLE 2. SUMMARY OF CALIBRATION PROCEDURES --Continued

Parameter	Method	Calibration	Frequency	Acceptance Criteria	Corrective Action
		DFTPP Tuning Standard	Start of run, every 12 hours	Mass/Criterion: 51: 30-60% of mass 198 68: <2% of mass 69 69: (reference only) 70: <2% of mass 69 127: 40-60% of mass 198 197: <1% of mass 198 198: Base peak 199: 5-9% of mass 198 275: 10-30% of mass 198 365: >1% of mass 198 441: Present but <mass 443 442: >40% of mass 198 443: 17-23% of mass 442	1) Retune instrument 2) Reanalyze BFB
		Continuing Calibration Standard	Daily before sample5analysis; every 12 hours	Meet Min. Response Factors for SPCCs ≤ 30% difference for CCCs	1) Check peak assignments and integration 2) Evalute system 3) Reanalyze standard 4) Recalibrate if necessary
Semivolatile Organics	SW8270	Internal Standard	Every sample	Area = 50-200% of Cont. Cal. Standard	1) Check integration 2) Evaluate system 3) Assess impact on data 4) Reanalyze sample if required
Explosives	SW8330	Multipoint Calibration (minimum 5 points)	Initially and as required	% RSD ≤ 25%	1) Evaluate system 2) Recalibrate
		Daily standard	Daily, prior to analysis	75-125% recovery	1) Evaluate system 2) Reanalyze standard 3) Recalibrate if required

TABLE 2. SUMMARY OF CALIBRATION PROCEDURES --Continued

Parameter	Method	Calibration	Frequency	Acceptance Criteria	Corrective Action
		Continuing calibration check standard	Every 10 samples, end of run,	75-125% recovery	1) Evaluate system 2) assess impact on data 3) Reanalyze standard 4) Recalibrate if required
Dioxins and Furans	SW8280	Check tuning using FC43	As needed	Tune for max. sensitivity of m/z 414;m/z 502 ± 4-10% of m/z 219.	1) Retune instrument. 2) Reanalyze FC43.
		Window Defining Mix (WDM); can be combined with CPSM.	Prior to initial calibration and after adjustments or maintenance which could affect retention limits	None; used to set retention times.	1) Readjust windows 2) Reanalyze WDW/CPSM
		Column Performance Check Solution (CPSM)	Prior to initial and continuing calibrations (SP-2331 column only)	Resolution of TCDDs <25%)	1) Evaluate system 2) Perform maintenance 3) Reanalyze CPSM
		Multipoint Calibration (ICAL; 5 points)	Initially and as required	Refer to table 2A for ion abundance ratios. Internal standard S/N >10:1. Unlabelled PCDDs/ PCDFs S/N >2.5:1. RSD <15%. RTs consistent with WDW.	1) Evaluate system 2) Perform maintenance 3) Recalibrate
		Continuing calibration check standard (CCAL)	Once per 12 hours, prior to sample analysis	≤ 30% difference from the ICAL Relative Response Factors (RRFs)	1) Evaluate system 2) Reanalyze check standard 3) Recalibrate as necessary

TABLE 3. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES

Parameter	Method	QC Element	Frequency	Acceptance Criteria	Corrective Action
Common Anions	E300.0	Method Blank	1/batch	< Reporting Limit	1) Reanalyze method blank 2) Assess impact on data 3) Rerun samples if required
		MS	5%	Refer to Table 4	1) Check calculations 2) Analyze DCS 3) Assess impact on data 4) Rerun samples if required
		MSD	5%	Refer to Table 4	Same as MS
		DCS	1 pair/batch	Refer to Table 4	1) Check calculations 2) Reanalyze DCS 3) Verify calibration 4) Assess impact on data 5) Rerun samples if required
pH	SW9040 (w), SW9045 (s)	DCS	1 pair/batch	Refer to Table 4	1) Reanalyze DCS 2) Recalibrate if required 3) Reanalyze affected samples
Specific Conductance	E120.1	DCS	1 pair/batch	Refer to Table 4	1) Reanalyze DCS 2) Recalibrate if required 3) Reanalyze affected samples
Moisture Content	D2216	None	N/A	N/A	N/A
Total Dissolved Solids (TDS)	E160.1	Method Blank	1/batch	Weight change between -0.5 and 2.0 mg	1) Redry and reweigh samples 2)Clean all apparatus 3) Assess impact on data 4) Reanalyze samples if required

TABLE 3. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES --Continued

Parameter	Method	QC Element	Frequency	Acceptance Criteria	Corrective Action
		DCS	1 pair/batch	Refer to Table 4	1) Check calculations 2) Redry and reweigh samples 3) Assess impact on data 4) Reanalyze samples if required
		MS	5%	Refer to Table 4	1) Check calculations 2) Redry and reweigh samples 3) Check DCS results 4) Assess impact on data 5) Reanalyze samples if required
		MSD	5%	Refer to Table 4	Same as Ms
		TDS/Conductance Ratio	Every sample	0.55-0.81 (advisory only; not applicable to very low or very high TDS)	1) Check calculations 2) Check conductance results 3) Review other available data 4) Reanalyze sample only if lab error is suspected
Alkalinity	E310.1	Method Blank	1/batch	<Reporting Limit	1) Clean and maintain system 2) Assess impact on data 3) Reanalyze samples if required
		DCS	1 pair/batch	Refer to Table 4	1) Check calculations 2) Verify titrant concentration 3) Reanalyze samples if required

TABLE 3. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES --Continued

Parameter	Method	QC Element	Frequency	Acceptance Criteria	Corrective Action
		MS	5%	Refer to Table 4	1) Check calculations 2) Check DCS results 3) Assess impact on data 4) Reanalyze samples if required
		MSD	5%	Refer to Table 4	Same as MS
Metals, Total	SW7060, SW7421, SW7740, SW7841	Method Blank	1/batch	< Reporting Limit	1) Reanalyze blank 2) Assess impact on data 3) Reprep samples if required
		DCS	1 pair/batch	Refer to Table 4	1) Verify calibration 2) Reanalyze DCS 3) Assess impact on data 4) Reprep samples if required
		Analytical Spike	Every Sample	80-120% Recovery	1) If sample >/= RL, dilute and reanalyze 2) If sample < RL & rec. <40%, dilute and reanalyze 3) If sample < RL & rec. 40-80% Report ND at 2x RL 4) If sample < RL & rec. >120%, Report ND at 2x RL
		MS	5%	Refer to Table 4	1) Check calculations 2) Check DCS & spike recovery 3) Assess impact on data 4) Reprep samples if required
		MSD	5%	Refer to Table 4	Same as MS

TABLE 3. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES --Continued

Parameter	Method	QC Element	Frequency	Acceptance Criteria	Corrective Action
	SW7470 (w), SW7471 (s)	Method Blank	1/Batch	≤ Reporting Limit	1) Reanalyze blank 2) Assess impact on data 3) Reprep samples if required
Metals, Total		DCS	1 pair/batch	Refer to Table 4	1) Verify calibration 2) Reanalyze DCS 3) Assess impact on data 4) Reprep samples if required
	SW7470 (w), SW7471 (s)	MS	5%	Refer to Table 4	1) Check calculations 2) Check DCS recovery 3) Assess impact on data 4) Reprep samples if required
		MSD	5%	Refer to Table 4	Same as MS
	SW6010	Method blank	1/batch	≤ Reporting Limit	1) Reanalyze blank 2) Assess impact on data 3) Reprep samples if required
		DCS	1 pair/batch	Refer to Table 4	1) Verify calibration 2) Reanalyze DCS 3) Assess impact on data 4) Reprep samples if required
		MS	5%	Refer to Table 4	1) Check calculations 2) Check DCS results 3) Assess impact on data 4) Reprep samples if required
		MSD	5%	Refer to Table 4	Same as MS

TABLE 3. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES --Continued

Parameter	Method	QC Element	Frequency	Acceptance Criteria	Corrective Action
Pesticides and PCBs	SW8080	Method Blank	1/batch	< Reporting Limit	1) Reanalyze method blank 2) Assess impact on data 3) Reextract samples if required
		MS	5%	Refer to Table 4	1) Check calculations 2) Reanalyze MS/MSD 3) Check surrogate, DCS results 4) Reextract samples if required
		MSD	5%	Refer to Table 4	Same as MS
		DCS	1 pair/batch	Refer to Table 4	1) Check calculations 2) Reanalyze DCS 3) Assess impact on data 4) Reextract samples if required
		SCS	1/batch	Refer to Table 4	1) Check calculations 2) Reanalyze SCS 3) Check DCS, MS/MSD results
		Surrogate Spike	Every sample	Refer to Table 4	1) Check calculations 2) Reanalyze sample 3) Assess impact on data 4) Reextract sample if required
Herbicides	SW8150	Methylation Standard	1/batch	> 80% Recovery	1) Check calculations 2) Check preparation method, reagents 3) Assess impact on data 4) Reextract samples if required

TABLE 3. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES --Continued

Parameter	Method	QC Element	Frequency	Acceptance Criteria	Corrective Action
		Method blank	1/batch	< Reporting Limit	1) Reanalyze method blank 2) Assess impact on data 3) Reextract samples if required
		MS	5%	Refer to Table 4	1) Check calculations 2) Reanalyze MS/MSD 3) Check surrogate, DCS results 4) Assess impact on data 5) Reextract samples if required
		MSD	5%	Refer to Table 4	Same as MS
		DCS	1 pair/batch	Refer to Table 4	1) Check calculations 2) Reanalyze DCS 3) Assess impact on data 5) Reextract samples if required
		SCS	1/batch	Refer to Table 4	1) Check calculations 2) Reanalyze SCS 3) Check DCS, MS/MSD results
		Surrogate Spike	Every sample	Refer to Table 4	1) Check calculations 2) Reanalyze sample 3) Assess impact on data 4) Reextract sample if required
Volatile Organics	SW8240	Method Blank	1/batch	< Reporting Limit except Methylene Chloride, acetone, 2-Butanone which must be <5x Reporting Limit	1) Reanalyze method blank 2) Assess impact on data 3) Reanalyze samples if required

TABLE 3. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES --Continued

Parameter	Method	QC Element	Frequency	Acceptance Criteria	Corrective Action
		MS	5%	Refer to Table 4	1) Check calculations 2) Reanalyze MS/MSD 3) Check surrogate, DCS results 4) Assess impact on data 4) Reanalyze samples if required
		MSD	5%	Refer to Table 4	Same as MS
		DCS	1 pair/batch	Refer to Table 4	1) Check calculations 2) Reanalyze DCS 3) Assess impact on data 4) Reanalyze samples if required
		SCS	1/batch	Refer to Table 4	1) Check calculations 2) Reanalyze SCS 3) Check DCS, MS/MSD results
		Surrogate Spike	Every sample	Refer to Table 4	1) Check calculations 2) Assess impact on data 3) Reanalyze sample if required
Semivolatile Organics	SW8270	Method Blank	1/batch	< Reporting Limit (Phthalate Esters <5x Reporting Limit)	1) Reanalyze method blank 2) Assess impact on data 3) Reextract samples if required
		MS/MSD	1 pair/20 samples	Refer to Table 4	1) Check calculations 2) Reanalyze MS/MSD 3) Check surrogate, DCS results 4) Assess impact on data 5) Reanalyze samples if required

TABLE 3. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES --Continued

Parameter	Method	QC Element	Frequency	Acceptance Criteria	Corrective Action
		DCS	1 pair/batch	Refer to Table 4	1) Check calculations 2) Reanalyze DCS 3) Assess impact on data 5) Reanalyze samples if required
		SCS	1/batch	Refer to Table 4	1) Check calculations 2) Reanalyze SCS 3) Check DCS, MS/MSD results
		Surrogate Spike	Every sample, DCS, MS, MSD, Method Blank	Refer to Table 4	1) Check calculations 2) Assess impact on data 3) Reanalyze sample if required
Explosives	SW8330	Method Blank	1/batch	< Reporting Limit	1) Reanalyze method blank 2) Assess impact on data 3) Reextract samples if required
		MS	5%	Refer to Table 4	1) Check calculations 2) Check DCS results 3) Assess impact on data 4) Reextract samples if required
		MSD	5%	Refer to Table 4	Same as MS
		DCS	1 pair/batch	Refer to Table 4	1) Check calculations 2) Verify calibration 3) Reanalyze DCS 4) Assess impact on data 5) Reextract samples if required

TABLE 3. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES --Continued

Parameter	Method	QC Element	Frequency	Acceptance Criteria	Corrective Action
Dioxins and Furans	SW8280	Method Blank	1 per analytical batch, not to exceed 20 samples per batch	< Target Detection Limit	1) Run system blank 2) Assess impact on data 3) Reanalyze blank 4) Reprep batch as necessary
		Internal Standards	Every sample, method blank, and DCS.	Recovery within limits in table 4 or S/N > 10:1	1) Recalculate S/N ratio 2) Reanalyze extract 3) Reextract sample using smaller aliquot
		MS/MSD	1 set per 20 samples	Refer to table 4	1) Check calculations 2) Assess impact on data 3) Reanalyze once 4) Reextract if necessary 5) Qualify data
		DCS	1 pair per batch, not to exceed 20 samples	Refer to table 4	1) Check calculations 2) Assess impact on data 3) Reanalyze once 4) Reextract if necessary
		Recovery Standard	Every sample, method blank, and DCS prior to instrument analysis.	Refer to table 2A	1) Check calculations 2) Reanalyze

TABLE 4. ACCEPTANCE CRITERIA FOR QC SAMPLES

Parameter	Analytical Method	QC Element	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil (mg/kg)	Laboratory-Established Control Limits Recovery (%) Water	Recovery (%) Soil	RPD (%) Water	RPD (%) Soil
Common Anions	E300.0	DCS	Fluoride	5.0	100	91-112	91-112	10	10
			Chloride	10.0	500	90-110	92-113	10	10
			Nitrate	20.0	10.0	91-113	91-111	10	10
			Phosphate	20.0	100	94-116	91-113	10	14
			Sulfate	200	250	90-110	91-111	10	10
		MS/MSD	Fluoride	5.0	100	91-112	91-112	10	10
			Chloride	10.0	500	90-110	92-113	10	10
			Nitrate	20.0	10.0	91-113	91-111	10	10
			Phosphate	20.0	100	94-116	91-113	10	14
			Sulfate	200	250	90-110	91-111	10	10
pH	SW9040 (w)	DCS	pH	9.1	N/A	97-102	N/A	10	N/A
	SW9045 (s)	DCS	pH	N/A	9.1	N/A	98-102	N/A	5
Specific Conductance	E120.1	DCS	Specific Conductance at 25 °C	1	1,650	90-110	90-110	10	20
Total Dissolved Solids (TDS)	E160.1	DCS	Total Dissolved Solids	1,400	N/A	87-107	N/A	10	N/A
		MS/MSD	Total Dissolved Solids	1,400	N/A	87-107	N/A	10	N/A
Alkalinity	E310.1	DCS	Alkalinity as $CaCO_3$	211	1,280	91-111	90-110	10	10
		MS/MSD	Alkalinity as $CaCO_3$	211	1,280	91-111	90-110	10	10
Metals, Total	SW7060	DCS	Arsenic	0.030	3.0	81-116	75-128	13	13
	SW7421	DCS	Lead	0.030	3.0	71-136	71-132	17	16
	SW7470	DCS	Mercury	0.0010	N/A	83-112	N/A	12	N/A
	SW7471	DCS	Mercury	N/A	0.10	N/A	88-113	N/A	12
	SW7740	DCS	Selenium	0.030	3.0	73-125	71-129	15	12
	SW7841	DCS	Thallium	0.030	3.0	75-125	75-125	20	20

TABLE 4. ACCEPTANCE CRITERIA FOR QC SAMPLES --Continued

Parameter	Analytical Method	QC Element	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil (mg/kg)	Laboratory-Established Control Limits Recovery (%) Water	Recovery (%) Soil	RPD (%) Water	RPD (%) Soil
	SW6010	DCS	Aluminum	0.20	20	80-116	80-120	10	20
			Antimony	0.50	50	80-115	80-120	14	20
			Barium	2.00	200	80-114	80-120	10	20
			Beryllium	0.050	5.0	80-120	80-120	10	20
			Cadmium	0.050	5.0	80-119	80-120	16	20
			Calcium	100	10,000	80-114	80-120	10	20
			Chromium	0.20	20	80-116	80-120	11	20
			Cobalt	0.50	50	80-114	80-120	10	20
			Copper	0.25	25	80-120	80-120	10	20
			Iron	1.00	100	80-120	80-120	11	20
			Magnesium	50	5,000	81-120	80-120	10	20
			Nickel	0.50	50	80-114	80-120	10	20
			Potassium	50	50,000	80-120	80-120	13	20
			Silver	0.050	5.0	80-119	80-120	15	20
			Sodium	100	10,000	80-120	80-120	10	20
			Tin	10	1,000	80-120	80-120	20	20
			Vanadium	0.50	50	80-116	80-120	10	20
			Zinc	0.50	50	80-120	80-120	13	20
Metals. Total	SW7060	MS/MSD	Arsenic	0.030	3.0	81-116	75-128	13	13
Metals, Total	SW7421	MS/MSD	Lead	0.030	3.0	71-136	71-132	17	16
Metals, Total	SW7470	MS/MSD	Mercury	0.0010	N/A	83-112	N/A	12	N/A
Metals. Total	SW7471	MS/MSD	Mercury	N/A	0.10	N/A	88-113	N/A	12
Metals. Total	SW7740	MS/MSD	Selenium	0.030	3.0	73-125	71-129	15	12
Metals, Total	SW7841	MS/MSD	Thallium	0.030	99	75-125	48-152	20	20
Metals. Total	SW6010	MS/MSD	Aluminum	0.20	20	80-120	80-120	20	20
			Antimony	0.50	50	80-120	80-120	20	20

TABLE 4. ACCEPTANCE CRITERIA FOR QC SAMPLES --Continued

Parameter	Analytical Method	QC Element	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil (mg/kg)	Laboratory-Established Control Limits Recovery (%) Water	Recovery (%) Soil	RPD (%) Water	RPD (%) Soil
			Barium	2.00	200	80-120	80-120	20	20
			Beryllium	0.050	5.0	80-120	80-120	20	20
			Cadmium	0.050	5.0	80-120	80-120	20	20
			Calcium	100	10,000	80-120	80-120	20	20
			Chromium	0.50	50	80-120	80-120	20	20
			Cobalt	0.50	50	80-120	80-120	20	20
			Copper	0.25	25	80-120	80-120	20	20
			Iron	1.00	100	80-120	80-120	20	20
			Magnesium	50	5,000	80-120	80-120	20	20
			Nickel	0.50	50	80-120	80-120	20	20
			Potassium	50	5,000	80-120	80-120	20	20
			Silver	0.050	5.0	80-120	80-120	20	20
			Sodium	100	10,000	80-120	80-120	20	20
			Tin	10	1,000	80-120	80-120	20	20
			Vanadium	0.50	50	80-120	80-120	20	20
			Zinc	0.50	50	80-120	80-120	20	20
Pesticides and PCBs	SW8080	DCS	gamma-BHC (Lindane)	0.20	26.7	81-117	63-130	13	11
			Heptachlor	0.20	26.7	72-125	62-136	11	10
			Aldrin	0.20	26.7	69-112	71-122	16	11
			Dieldrin	0.50	66.7	77-111	57-123	13	10
			Endrin	0.50	66.7	83-122	58-133	14	10
			4,4'-DDT	0.50	66.7	76-125	63-137	14	10
		MS/MSD	gamma-BHC (Lindane)	0.20	27	32-127	32-127	20	20
			Heptachlor	0.20	27	34-111	34-111	20	20
			Aldrin	0.20	27	42-122	42-122	20	20
			Dieldrin	0.50	67	36-146	36-146	20	20

TABLE 4. ACCEPTANCE CRITERIA FOR QC SAMPLES --Continued

Parameter	Analytical Method	QC Element	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil (mg/kg)	Laboratory-Established Control Limits Recovery (%) Water	Recovery (%) Soil	RPD (%) Water	RPD (%) Soil
			Endrin	0.50	67	30-147	30-147	20	20
			4,4'-DDT	0.50	67	25-160	25-160	20	20
		SCS	Dibutyl chlorendate	1.00	66.7	56-138	42-154	N/A	N/A
		Surrogate	Tetrachloro-m-xylene	N/A	8.33	N/A	30-150	N/A	N/A
			Dibutyl chlorendate	1.00	67	56-138	42-154	N/A	N/A
			Decachlorobiphenyl	N/A	8.33	N/A	30-150	N/A	N/A
Herbicides	SW8150	DCS	2,4-D	5.00	100	44-97	37-100	34	36
			2,4,5-TP (Silvex)	1.00	20.0	49-102	42-107	32	29
			2,4,5-T	1.00	20.0	47-110	29-118	32	29
		MS/MSD	2,4-D	5.00	100	44-97	37-100	34	36
			2,4,5-TP (Silvex)	1.00	20.0	49-102	42-107	32	29
			2,4,5-T	1.00	20.0	47-110	29-118	32	29
		SCS	DCAA	5.00	100	45-123	42-125	N/A	N/A
			2,4-DB	5.00	170	60-120	60-120	N/A	N/A
		Surrogate	DCAA	5.00	100	45-123	42-125	N/A	N/A
Volatile Organics	SW8240	DCS	1,1-Dichloroethene	50.0	50.0	74-124	65-137	17	20
			Trichloroethene	50.0	50.0	77-119	83-118	13	12
			Benzene	50.0	50.0	80-117	80-119	12	10
			Toluene	50.0	50.0	80-119	80-119	11	12
			Chlorobenzene	50.0	50.0	81-120	80-119	14	12
		MS/MSD	1,1-Dichloroethene	50.0	50.0	1-234	1-234	17	20
			Trichloroethene	50.0	50.0	71-157	71-157	13	12
			Benzene	50.0	50.0	37-151	37-151	12	10
			Toluene	50.0	50.0	47-150	47-150	11	12
			Chlorobenzene	50.0	50.0	37-160	37-160	14	12
		SCS	1,2-Dichloroethane-d4	50.0	50.0	85-111	82-112	N/A	N/A

TABLE 4. ACCEPTANCE CRITERIA FOR QC SAMPLES --Continued

Parameter	Analytical Method	QC Element	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil (mg/kg)	Laboratory-Established Control Limits Recovery (%) Water	Recovery (%) Soil	RPD (%) Water	RPD (%) Soil
			4-Bromofluorobenzene	50.0	50.0	86-110	84-109	N/A	N/A
			Toluene-d8	50.0	50.0	91-110	90-112	N/A	N/A
		Surrogate	1,2-Dichloroethane-d4	50.0	50.0	88-110	81-117	N/A	N/A
			4-Bromofluorobenzene	50.0	50.0	86-115	74-121	N/A	N/A
			Toluene-d8	50.0	50.0	86-115	70-121	N/A	N/A
Semivolatile Organics	SW8270	DCS	Phenol	100	6,670	45-109	45-107	29	19
			2-chlorophenol	100	6,670	47-111	46-112	29	17
			1,4-dichlorobenzene	50.0	3,330	32-103	58-101	28	22
			N-nitroso-di-n-propylamine	50.0	3,330	49-107	58-101	24	18
			1,2,4-trichlorobenzene	50.0	3,330	44-102	59-103	27	24
			4-chloro-3-methylphenol	100	6,670	50-115	41-123	27	16
			Acenaphthene	50.0	3,330	47-109	54-110	24	15
			4-Nitrophenol	100	6,670	40-127	30-132	51	22
			2,4-Dinitrotoluene	50.0	3,330	46-118	51-117	22	17
			Pentachlorophenol	100	6,670	30-136	32-130	34	29
			Pyrene	50.0	3,330	52-115	52-115	23	20
		MS/MSD	Phenol	100	6,670	5-112	5-112	29	19
			2-chlorophenol	100	6,670	23-134	23-134	29	17
			1,4-dichlorobenzene	50.0	3,330	20-124	20-124	28	22
			N-nitroso-di-n-propylamine	50.0	3,330	1-230	1-230	24	18
			1,2,4-trichlorobenzene	50.0	3,330	44-142	44-142	27	24
			4-chloro-3-methylphenol	100	6,670	22-127	22-127	27	16
			Acenaphthene	50.0	3,330	47-145	47-145	24	15
			4-Nitrophenol	100	6,670	1-132	1-132	51	22

TABLE 4. ACCEPTANCE CRITERIA FOR QC SAMPLES --Continued

Parameter	Analytical Method	QC Element	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil (mg/kg)	Laboratory-Established Control Limits Recovery (%) Water	Recovery (%) Soil	RPD (%) Water	RPD (%) Soil
			2,4-Dinitrotoluene	50.0	3,330	39-139	39-139	22	17
			Pentachlorophenol	100	6,670	14-176	14-176	34	29
			Pyrene	50.0	3,330	52-115	52-115	23	20
		SCS	Nitrobenzene-d5	100	1,670	49-113	62-110	N/A	N/A
			2-Fluorobiphenyl	100	1,670	43-104	61-114	N/A	N/A
			Terphenyl-d14	100	1,670	33-139	49-137	N/A	N/A
			2-Fluorophenol	200	3,330	42-100	60-115	N/A	N/A
			Phenol-d5	200	3,330	50-94	61-111	N/A	N/A
			2,4,6-Tribromophenol	200	3,330	33-123	44-110	N/A	N/A
		Surrogate	Nitrobenzene-d5	100	1,670	35-114	23-120	N/A	N/A
			2-Fluorobiphenyl	100	1,670	43-116	30-115	N/A	N/A
			Terphenyl-d14	100	1,670	33-141	18-137	N/A	N/A
			2-Fluorophenol	200	3,330	10-94	24-113	N/A	N/A
			Phenol-d5	200	3,330	21-100	25-121	N/A	N/A
			2,4,6-Tribromophenol	200	3,330	10-123	19-122	N/A	N/A
Explosives	SW8330 (HPLC)	DCS	HMX	5.0	1.0	60-132	70-138	30	17
			RDX	5.0	1.0	57-131	63-147	33	18
			1,3,5-Trinitrobenzene	5.0	1.0	42-97	59-133	40	15
			1,3-Dinitrobenzene	5.0	1.0	43-132	65-152	40	16
			Tetryl	5.0	1.0	1-109	1-131	40	50
			Nitrobenzene	5.0	1.0	7-117	60-159	40	20
			2,4,6-Trinitrotoluene	5.0	1.0	17-93	68-122	40	17
			4-Amino-2,6-dinitrotoluene	5.0	1.0	49-91	68-157	40	28
			2-Amino-4,6-dinitrotoluene	2.5	0.5	51-98	63-142	40	20
			2,6-Dinitrotoluene	5.0	1.0	40-115	53-150	40	24
			2,4-Dinitrotoluene	2.5	0.5	53-110	65-149	40	18

TABLE 4. ACCEPTANCE CRITERIA FOR QC SAMPLES --Continued

Parameter	Analytical Method	QC Element	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil (mg/kg)	Laboratory-Established Control Limits Recovery (%) Water	Recovery (%) Soil	RPD (%) Water	RPD (%) Soil
			2-Nitrotoluene	5.0	1.0	41-95	63-149	40	19
			4-Nitrotoluene	5.0	1.0	40-91	73-144	40	21
			3-Nitrotoluene	5.0	1.0	45-96	34-149	40	21
		MS/MSD	HMX	5.0	1.0	60-132	70-138	40	17
			RDX	5.0	1.0	57-131	63-147	33	18
			1,3,5-Trinitrobenzene	5.0	1.0	42-97	59-133	40	15
			1,3-Dinitrobenzene	5.0	1.0	43-132	65-152	40	16
			Tetryl	5.0	1.0	1-109	1-131	40	50
			Nitrobenzene	5.0	1.0	7-117	60-159	40	20
			2,4,6-Trinitrotoluene	5.0	1.0	17-93	68-122	40	17
			4-Amino-2,6-dinitrotoluene	5.0	1.0	49-91	68-157	40	28
			2-Amino-4,6-dinitrotoluene	2.5	0.5	51-98	63-142	40	20
			2,6-Dinitrotoluene	5.0	1.0	40-115	53-150	40	24
			2,4-Dinitrotoluene	2.5	0.5	53-110	65-149	40	18
			2-Nitrotoluene	5.0	1.0	41-95	63-149	40	19
			4-Nitrotoluene	5.0	1.0	40-91	73-144	40	21
			3-Nitrotoluene	5.0	1.0	45-96	34-149	40	21
Dioxins and Rurans	SW8280	Internal Standards (d,f)	13C-2,3,7,8-TCDF	25	25	40-120	40-120	N/A	N/A
			13C-2,3,7,8-TCDD	25	25	40-120	40-120	N/A	N/A
			13C-PeCDD	50	50	40-120	40-120	N/A	N/A
			13C-HxCDD	50	50	40-120	40-120	N/A	N/A
			13C-HpCDD	50	50	40-120	40-120	N/A	N/A
			13C-OCDD	125	125	40-120	40-120	N/A	N/A
		MS/MSD	2,3,7,8-TCDF	10	10	72-136	60-139	50	N/A
			2,3,4,7,8-PeCDF	10	10	74-110	58-131	50	N/A
			1,2,3,4,7,8-HxCDF	10	10	80-121	60-128	50	N/A

TABLE 4. ACCEPTANCE CRITERIA FOR QC SAMPLES --Continued

Parameter	Analytical Method	QC Element	Spiking Compounds	Spike Concentration Water (mg/L)	Spike Concentration Soil (mg/kg)	Laboratory-Established Control Limits Recovery (%) Water	Recovery (%) Soil	RPD (%) Water	RPD (%) Soil
			1,2,3,4,6,7,8-HpCDF	10	10	61-147	55-139	50	N/A
			OCDF (d)	50	50	50-150	50-150	50	N/A
			2,3,7,8-TCDD	10	10	76-122	72-121	50	N/A
			1,2,3,7,8-PeCDD	10	10	71-116	63-134	50	N/A
			1,2,3,4,7,8-HxCDD	10	10	80-112	64-120	50	N/A
			1,2,3,4,6,7,8-HpCDD	10	10	69-133	62-134	50	N/A
			OCDD (d)	50	50	50-150	50-150	50	N/A
		DCS	2,3,7,8-TCDF	10	10	72-136	60-139	50	N/A
			2,3,4,7,8-PeCDF	10	10	74-110	58-131	50	N/A
			1,2,3,4,7,8-HxCDF	10	10	80-121	60-128	50	N/A
			1,2,3,4,6,7,8-HpCDF	10	10	61-147	55-139	50	N/A
			OCDF (d)	50	50	50-150	50-150	50	N/A
			2,3,7,8-TCDD	10	10	76-122	72-121	50	N/A
			1,2,3,7,8-PeCDD	10	10	71-116	63-134	50	N/A
			1,2,3,4,7,8-HxCDD	10	10	80-112	64-120	50	N/A
			1,2,3,4,6,7,8-10	10	10	69-133	62-134	50	N/A
			OCDD (d)	50	50	50-150	50-150	50	N/A

(d) Method default limits. Signal-to-noise ratio is also evaluated for data acceptability.
(f) These labelled analytes are spiked into all samples.

APPENDIX C

PROJECT DETECTION LIMITS

Appendix C
Project Detection Limits

Volatile Organics; Appendix IX List

Component	Method	Reporting Limit Water (µg/L)	Reporting Limit Soil (µg/kg)
Acetone	SW8240	10	10
Acetonitrile	SW8240	200	200
Acrolein	SW8240	100	100
Acrylonitrile	SW8240	100	100
Allyl chloride	SW8240	10	10
Benzene	SW8240	5	5
Bromodichloromethane	SW8240	5	5
Bromoform	SW8240	5	5
Bromomethane	SW8240	10	10
2-Butanone (MEK)	SW8240	10	10
Carbon disulfide	SW8240	5	5
Carbon tetrachloride	SW8240	5	5
Chlorobenzene	SW8240	5	5
Chloroethane	SW8240	10	10
Chloroform	SW8240	5	5
Chloromethane	SW8240	10	10
Chloroprene	SW8240	5	5
Dibromochloromethane	SW8240	5	5
"1,2-Dibromo-3-chloro-propane (DBCP)"	SW8240	10	10
"1,2-Dibromoethane (EDB)"	SW8240	10	10
Dibromomethane	SW8240	5	5
"trans-1,4-Dichloro-2-butene"	SW8240	5	5
Dichlorodifluoromethane	SW8240	20	20
"1,1-Dichloroethane"	SW8240	5	5
"1,2-Dichloroethane"	SW8240	5	5
"1,1-Dichloroethene"	SW8240	5	5
"1,2-Dichloroethene (total)"	SW8240	5	5
"1,2-Dichloropropane"	SW8240	5	5
"cis-1,3-Dichloropropene"	SW8240	5	5
"trans-1,3-Dichloropropene"	SW8240	5	5
"1,4-Dioxane"	SW8240	500	500
Ethylbenzene	SW8240	5	5
Ethyl methacrylate	SW8240	20	20
"1,1,1,2-Tetrachloroethane"	SW8240	5	5
"1,1,1-Trichloroethane"	SW8240	5	5
"1,1,2,2-Tetrachloroethane"	SW8240	5	5
"1,1,2-Trichloroethane"	SW8240	5	5
"1,2,3-Trichloropropane"	SW8240	5	5
2-Hexanone	SW8240	10	10
4-Methyl-2-pentanone (MIBK)	SW8240	10	10
Iodomethane	SW8240	5	5
Isobutanol	SW8240	200	200
Methacrylonitrile	SW8240	5	5
Methyl methacrylate	SW8240	20	20

Component	Method	Reporting Limit Water (µg/L)	Reporting Limit Soil (µg/kg)
Methylene chloride	SW8240	5	5
Propionitrile	SW8240	5	5
Styrene	SW8240	5	5
Tetrachloroethene	SW8240	5	5
Toluene	SW8240	5	5
Trichloroethene	SW8240	5	5
Trichlorofluoromethane	SW8240	5	5
Vinyl acetate	SW8240	10	10
Vinyl chloride	SW8240	10	10
Xylenes (total)	SW8240	5	5

Organochlorine Pesticides and PCBs; Appendix IX List

Component	Method	Reporting Limit Water (µg/L)	Reporting Limit Soil (µg/kg)
Aldrin	SW8080	0.05	1.7
Aroclor 1016	SW8080	1	33
Aroclor 1221	SW8080	1	33
Aroclor 1232	SW8080	1	33
Aroclor 1242	SW8080	1	33
Aroclor 1248	SW8080	1	33
Aroclor 1254	SW8080	1	33
Aroclor 1260	SW8080	1	33
alpha-BHC	SW8080	0.05	1.7
beta-BHC	SW8080	0.05	1.7
delta-BHC	SW8080	0.05	1.7
gamma-BHC (Lindane)	SW8080	0.05	1.7
alpha-Chlordane	SW8080	0.05	1.7
gamma-Chlordane	SW8080	0.05	1.7
Chlorobenzilate	SW8080	0.1	3.3
"4,4'-DDD"	SW8080	0.1	3.3
"4,4'-DDE"	SW8080	0.1	3.3
"4,4'-DDT"	SW8080	0.1	3.3
Diallate	SW8080	1	33
Dieldrin	SW8080	0.1	3.3
Endosulfan I	SW8080	0.05	1.7
Endosulfan II	SW8080	0.1	3.3
Endosulfan sulfate	SW8080	0.1	3.3
Endrin	SW8080	0.1	3.3
Endrin aldehyde	SW8080	0.1	3.3
Heptachlor	SW8080	0.05	1.7
Heptachlor epoxide	SW8080	0.05	1.7
Isodrin	SW8080	0.1	3.3
Kepone	SW8080	1	33
Methoxychlor	SW8080	0.5	17
Toxaphene	SW8080	5	170

Herbicides; Appendix IX List

Component	Method	Reporting Limit Water (µg/L)	Reporting Limit Soil (µg/kg)
"2,4-D"	SW8150	1.2	24
"2,4,5-TP (Silvex)"	SW8150	0.17	3.4
"2,4,5-T"	SW8150	0.2	4

Semivolatile Organics; Appendix IX List

Component	Method	Reporting Limit Water (µg/L)	Reporting Limit Soil (µg/kg)
Acenaphthene	SW8270	10	330
Acenaphthylene	SW8270	10	330
Acetophenone	SW8270	10	330
2-Acetylaminofluorene	SW8270	100	3,300
4-Aminobiphenyl	SW8270	10	330
Aniline	SW8270	10	330
Anthracene	SW8270	10	330
Aramite	SW8270	10	330
Benzo(a)anthracene	SW8270	10	330
Benzo(b)fluoranthene	SW8270	10	330
Benzo(k)fluoranthene	SW8270	10	330
"Benzo(g,h,i)perylene"	SW8270	10	330
Benzo(a)pyrene	SW8270	10	330
Benzyl alcohol	SW8270	10	330
4-Bromophenyl phenyl ether	SW8270	10	330
Butyl benzyl phthalate	SW8270	10	330
"2-sec-Butyl-4,6-dinitro-phenol"	SW8270	10	330
4-Chloroaniline	SW8270	10	330
bis(2-Chloroethoxy)methane	SW8270	10	330
bis(2-Chloroethyl) ether	SW8270	10	330
bis(2-Chloroisopropyl) ether	SW8270	10	330
4-Chloro-3-methylphenol	SW8270	10	330
2-Chloronaphthalene	SW8270	10	330
2-Chlorophenol	SW8270	10	330
4-Chlorophenyl phenyl ether	SW8270	10	330
Chrysene	SW8270	10	330
"Dibenz(a,h)anthracene"	SW8270	10	330
Dibenzofuran	SW8270	10	330
Di-n-butyl phthalate	SW8270	10	330
"1,2-Dichlorobenzene"	SW8270	10	330
"1,3-Dichlorobenzene"	SW8270	10	330
"1,4-Dichlorobenzene"	SW8270	10	330
"3,3'-Dichlorobenzidine"	SW8270	20	660
"2,4-Dichlorophenol"	SW8270	10	330
"2,6-Dichlorophenol"	SW8270	10	330
Diethyl phthalate	SW8270	10	330
Dimethoate	SW8270	--	--

Component	Method	Reporting Limit Water (µg/L)	Reporting Limit Soil (µg/kg)
p-Dimethylaminoazobenzene	SW8270	10	330
"7,12-Dimethylbenz(a)anthracene"	SW8270	10	330
"3,3'-Dimethylbenzidine"	SW8270	10	330
"a,a-Dimethylphenethylamine"	SW8270	10	330
"2,4-Dimethylphenol"	SW8270	10	330
Dimethyl phthalate	SW8270	10	330
"1,3-Dinitrobenzene"	SW8270	10	330
"4,6-Dinitro-2-methylphenol"	SW8270	50	1,600
"2,4-Dinitrophenol"	SW8270	50	1,600
"2,4-Dinitrotoluene"	SW8270	10	330
"2,6-Dinitrotoluene"	SW8270	10	330
Di-n-octyl phthalate	SW8270	10	330
Diphenylamine	SW8270	10	330
Disulfoton	SW8270	50	1,600
bis(2-Ethylhexyl)phthalate	SW8270	10	330
Ethyl methanesulfonate	SW8270	10	330
Famphur	SW8270	--	--
Fluoranthene	SW8270	10	330
Fluorene	SW8270	10	330
Hexachlorobenzene	SW8270	10	330
Hexachlorobutadiene	SW8270	10	330
Hexachlorocyclopentadiene	SW8270	10	330
Hexachloroethane	SW8270	10	330
Hexachlorophene	SW8270	--	--
Hexachloropropene	SW8270	10	330
"Indeno(1,2,3-cd)pyrene"	SW8270	10	330
Isophorone	SW8270	10	330
Isosafrole	SW8270	20	660
Methapyrilene	SW8270	10	330
3-Methylcholanthrene	SW8270	10	330
Methyl methanesulfonate	SW8270	10	330
2-Methylnaphthalene	SW8270	10	330
Methyl parathion	SW8270	50	1,600
2-Methylphenol	SW8270	10	330
3/4-Methylphenol	SW8270	10	330
Naphthalene	SW8270	10	330
"1,4-Naphthoquinone"	SW8270	10	330
1-Naphthylamine	SW8270	10	330
2-Naphthylamine	SW8270	10	330
2-Nitroaniline	SW8270	50	1,600
3-Nitroaniline	SW8270	50	1,600
4-Nitroaniline	SW8270	50	1,600
Nitrobenzene	SW8270	10	330
2-Nitrophenol	SW8270	10	330
4-Nitrophenol	SW8270	50	1,600
4-Nitroquinoline-1-oxide	SW8270	--	--
N-Nitroso-di-n-butylamine	SW8270	10	330
N-Nitrosodiethylamine	SW8270	10	330
N-Nitrosodimethylamine	SW8270	10	330
N-Nitrosodiphenylamine	SW8270	10	330

Component	Method	Reporting Limit Water (µg/L)	Reporting Limit Soil (µg/kg)
N-Nitroso-di-n-propylamine	SW8270	10	330
N-Nitrosomethylethylamine	SW8270	10	330
N-Nitrosomorpholine	SW8270	10	330
N-Nitrosopiperidine	SW8270	10	330
N-Nitrosopyrrolidine	SW8270	10	330
5-Nitro-o-toluidine	SW8270	10	330
Parathion	SW8270	50	1,600
Pentachlorobenzene	SW8270	10	330
Pentachloroethane	SW8270	10	330
Pentachloronitrobenzene	SW8270	50	1,600
Pentachlorophenol	SW8270	50	1,600
Phenacetin	SW8270	10	330
Phenanthrene	SW8270	10	330
Phenol	SW8270	10	330
4-Phenylenediamine	SW8270	--	--
Phorate	SW8270	100	3,300
2-Picoline	SW8270	10	330
Pronamide	SW8270	10	330
Pyrene	SW8270	10	330
Pyridine	SW8270	20	660
Safrole	SW8270	10	330
Sulfotepp	SW8270	50	1,600
"1,2,4,5-Tetrachlorobenzene"	SW8270	10	330
"2,3,4,6-Tetrachlorophenol"	SW8270	50	1,600
Thionazin	SW8270	50	1,600
2-Toluidine	SW8270	10	330
"1,2,4-Trichlorobenzene"	SW8270	10	330
"2,4,5-Trichlorophenol"	SW8270	50	1,600
"2,4,6-Trichlorophenol"	SW8270	10	330
"O,O,O-Triethylphosphorothioate"	SW8270	10	330
"1,3,5-Trinitrobenzene"	SW8270	10	330

Note: The list above includes the organophosphorus pesticides (method SW8140)

Dioxins and Furans; Appendix IX List

Component	Method	Reporting Limit Water (ng/L)	Reporting Limit Soil (ng/kg)
TCDFs (total)	SW8280	0.01	0.33
PeCDFs (total)	SW8280	0.01	0.33
HxCDFs (total)	SW8280	0.01	0.33
TCDDs (total)	SW8280	0.01	0.33
"2,3,7,8-TCDD"	SW8280	0.005	0.17
PeCDDs (total)	SW8280	0.01	0.33
HxCDDs (total)	SW8280	0.01	0.33

Note: Reporting limits for dioxins and furans are determined on a case-by-case basis. Target limits are given above.

Metals; Appendix IX List

Component	Method	Reporting Limit Water (mg/L)	Reporting Limit Soil (mg/kg)
Antimony	SW6010	0.06	6
Barium	SW6010	0.01	1
Beryllium	SW6010	0.002	0.2
Cadmium	SW6010	0.005	0.5
Chromium	SW6010	0.01	1
Cobalt	SW6010	0.01	1
Copper	SW6010	0.02	2
Nickel	SW6010	0.04	4
Silver	SW6010	0.01	1
Tin	SW6010	0.1	10
Vanadium	SW6010	0.01	1
Zinc	SW6010	0.02	2
Arsenic	SW7060	0.005	0.5
Lead	SW7421	0.005	0.5
Selenium	SW7740	0.005	0.5
Thallium	SW7841	0.005	0.5
Mercury	SW7470	0.0002	0.1

Explosives

Component	Method	Reporting Limit Water (µg/L)	Reporting Limit Soil (µg/kg)
HMX	SW8330	0.8	2.2
RDX	SW8330	0.84	1
"1,3,5-Trinitrobenzene"	SW8330	0.26	0.25
"1,3-Dinitrobenzene"	SW8330	0.11	0.25
Tetryl	SW8330	0.8	0.65
Nitrobenzene	SW8330	0.25	0.26
"2,4,6-Trinitrotoluene"	SW8330	0.11	0.25
"4-Amino-2,6-dinitrotoluene"	SW8330	0.06	0.25
"2-Amino-4,6-dinitrotoluene"	SW8330	0.035	0.25
"2,6-Dinitrotoluene"	SW8330	0.31	0.26
"2,4-Dinitrotoluene"	SW8330	0.02	0.25
2-Nitrotoluene	SW8330	0.25	0.25
4-Nitrotoluene	SW8330	0.25	0.25
3-Nitrotoluene	SW8330	0.25	0.25

Additional Analytes

Component	Method	Reporting Limit Water (mg/L)
Fluoride	E300.0	0.5
Chloride	E300.0	0.5
Nitrate as N	E300.0	0.5

Component	Method	Reporting Limit Water (mg/L)
Orthophosphate as P	E300.0	0.5
Sulfate	E300.0	0.5
Total Dissolved Solids	E160.1	10
"Alkalinity, Total as CaCO3 at pH 4.5"	E310.1	5
"Alkalinity, Bicarbonate as CaCO3 at pH 4.5"	E310.1	5
"Alkalinity, Carbonate as CaCO3 at pH 8.3"	E310.1	5
"Alkalinity, Hydroxide as Ca CO3"	E310.1	5
pH	SW9040	--
Specific Conductance at 25 C	E120.1	1 µmho/cm
Calcium	SW6010	5
Magnesium	SW6010	5
Potassium	SW6010	5
Sodium	SW6010	5

SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES*

Parameter	Method	Matrix	Container	Preservative	Maximum Holding Time
Volatile Organics	SW8240	Water	3 x 40 mL Vial	HCl to pH <2**	14 days
		Soil	4 oz. Glass	None	14 days
Semivolatile Organics	SW8270	Water	2 x 1 L Glass	None	7 days to extraction. 40 days extraction to analysis
		Soil	16 oz. Glass	None	14 days to extraction, 40 days extraction to analysis
Pesticides & PCBs	SW8080	Water	2 x 1 L Glass	None	7 days to extraction, 40 days extraction to analysis
		Soil	16 oz. Glass	None	14 days to extraction, 40 days extraction to analysis
Herbicides	SW8150	Water	1 L Glass	None	7 days to extraction, 40 days extraction to analysis
		Soil	16 oz. Glass	None	14 days to extraction, 40 days extraction to analysis
Explosives	SW8330	Water	1 L Glass	None	7 days to extraction, 40 days extraction to analysis
		Soil	16 oz. Glass	None	14 days to extraction, 40 days extraction to analysis
Metals, Total	SW6010,	Water	16 oz. Poly.	HNO3 to pH<2	6 months
	SW7060, SW7421, SW7740, SW7841	Soil	16 oz. Glass	None	6 months
Mercury, Total	SW7470	Water	16 oz. Poly.	HNO3 to pH <2	28 days
	SW7471	Soil	16 oz. Glass	None	28 days
Anions: Fluoride, Chloride, Sulfate	E300.0	Water	32 oz. Poly.	None	28 days
		Soil	8 oz. Glass	None	28 days after extraction
Nitrate, Phosphate	E300.0	Water	32 oz. Poly.	None	48 hours

SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES* --Continued

Parameter	Method	Matrix	Container	Preservative	Maximum Holding Time
		Soil	8 oz. Glass	None	48 hours after extraction
pH	SW9040	Water	32 oz. Poly.	None	As soon as possible
	SW9045	Soil	8 oz. Glass	None	As soon as possible after extraction
Specific Conductance	E120.1	Water	32 oz. Poly.	None	28 days
		Soil	8 oz. Glass	None	28 days after extraction
Moisture Content	D2216	Soil	8 or 16 oz. Glass	None	None established

* Holding times are calculated from the date of collection unless otherwise specified.

In addition to the preservative stated, all samples should be chilled to 4 °C.

** If samples contain residual chlorine, 4 drops of 10% sodium thiosulfate should be added.

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